

TABLE OF LENGTH EQUIVALENTS (METRIC SYSTEM)

10 millimeters (mm)	= 1 centimeter (cm)
10 centimeters	= 1 decimeter (dm)
10 decimeters	= 1 meter (m)
10 meters	= 1 dekameter (dkm)
10 dekameters	= 1 hectometer (hm)
10 hectometers	= 1 kilometer (km)

TABLE OF CAPACITY (VOLUME) EQUIVALENTS

10 milliliters (ml)	= 1 centiliter (cl)
10 centiliters	= 1 deciliter (dl)
10 deciliters	= 1 liter (l)
10 liters	= 1 dekaliter (dkl)
10 dekaliters	= 1 hectoliter (hl)
10 hectoliters	= 1 kiloliter (kl)

TABLE OF WEIGHT EQUIVALENTS

10 milligrams (mg)	= 1 centigram (cg)
10 centigrams	= 1 decigram (dg)
10 decigrams	= 1 gram (g)
10 grams	= 1 dekagram (dkg)
10 dekagrams	= 1 hectogram (hg)
10 hectograms	= 1 kilogram (kg)

TABLE OF METRIC—CUSTOMARY EQUIVALENTS

1 inch (in.)	= 2.54 cm	1 g	= 15.432 grains (gr.)
1 pound (lb)	= 453.6 g	1 ml	= 1.000027 cm ³
1 quart (qt)	= 0.946 l	1 fluid ounce (fl oz)	= 29.573 ml
		1 ml	= 16-20 drops
		1 atmosphere	= 760 mm Hg

Charles A. Brautlecht

THE LIBRARY
UNIVERSITY OF MAINE



GIFT OF
Dr. C.A. Brautlecht



Digitized by the Internet Archive
in 2026

[illegible]

LABORATORY MANUAL
IN
GENERAL CHEMISTRY

Laboratory Manual in General Chemistry

TO ACCOMPANY LINCOLN AND BANKS
GENERAL CHEMISTRY

BY
AZARIAH T. LINCOLN
PROFESSOR OF CHEMISTRY, CARLETON COLLEGE
AND
GEORGE B. BANKS
PROFESSOR OF PHYSICAL SCIENCE, NIAGARA UNIVERSITY



NEW YORK
PRENTICE-HALL, INC.
1928

COPYRIGHT, 1928, BY

PRENTICE-HALL, INC.

All rights reserved

PREFACE

This laboratory manual has been prepared primarily to accompany the authors' "General Chemistry." The page numbers given in parentheses following the titles of exercises refer to the pages in that text. It may be seen by a glance that the arrangement of the subject matter follows closely that of the text.

The text and the laboratory manual contain more material than can be covered in one year's work. Sufficient material has been included so that an instructor may make a selection suited to the particular type of course he prefers to offer. With this end in view, the manual is divided into three parts.

Part I comprises the exercises in general inorganic chemistry. There is enough material in this part to furnish the laboratory work for a whole year in elementary inorganic chemistry. Part II comprises the exercises in organic chemistry. These are provided for those instructors who desire to include in their course some organic chemistry. This may be the case, for example, with those who offer courses to agricultural and home economics students and give a more general course in beginning chemistry. The exercises in Part II can be substituted for some of those in Part I. Many teachers desire to devote a considerable portion of the second semester's laboratory work to qualitative analysis. This manual is arranged particularly for this purpose and in Part III are given directions for elementary systematic qualitative analysis. There has also been incorporated incomplete equations indicating the reactions taking place during the designated procedure. This is for the purpose of suggesting to the student what chemical changes are involved so that he can better understand the procedure as he studies it.

Since most of the graduated ware is now being marked in millimeters, all references to capacity are given in millimeters (ml) instead of cubic centimeters (cc.). It is customary to designate in grams the amounts of solid materials to be employed. This practice, of course, necessitates weighing. The authors have found that it is much more convenient to measure quantities by volume than by weight. Therefore, the quantities of solids

are expressed in milliliters. In parentheses immediately following, approximately equivalent quantities of the solids are expressed also in grams.

The concentrations of solutions to be used are stated in terms of normality or molarity. The methods of preparing some of the reagents are given in the Appendix.

We desire to express our gratitude for material and suggestions to those authors whose laboratory manuals we have consulted. Particularly do we wish to express our indebtedness to Mr. Warren K. Egloff, Niagara University, for checking over many of the exercises, for preparing drawings for the figures, and, especially, for reading the proof; to Mr. J. B. Cloke, Rensselaer Polytechnic Institute, for his assistance with the exercises in organic chemistry especially; to Mr. Lyle I. Gilbertson, formerly of Carleton College, for assisting with the qualitative procedure and equations; to Dr. F. F. Exner and Mr. Harold Klug, Carleton College, for their coöperation and assistance.

A. T. L.

G. B. B.

CONTENTS

Preface	PAGE V
List of Illustrations	xiii

INTRODUCTION

Laboratory Directions	2
Manipulation	3
I. Pouring Liquid from Containers	3
II. Transferring Dry Solids	3
III. Manipulating Glass Tubing	3
(a) To Cut Glass Tubing	3
(b) To Fire Polish Ends of Glass Tubing	3
(c) To Bend Glass Tubing	4
(d) To Draw or Seal a Glass Tube	4
IV. Heating	4
1. Water Bath	5
2. Steam Bath	5
3. Sand Bath	5
4. Air Bath	5
5. Oil and Molten Salts	5
6. Hot Plate	6
V. Evaporating	6
VI. Distilling	6
VII. Separating a Solid from a Liquid	6
1. Decantation	6
2. Filtration	6
(a) Folding the Filter Paper	7
(b) Introducing the Liquid onto a Filter Paper	7
(c) Removal of the Precipitate from a Beaker to a Filter Paper	7
VIII. Washing Precipitates	7
1. On Filter Paper	7
2. By Decantation	7
IX. Boring Stoppers	7
X. Constructing Apparatus	8
1. Fitting Glass Tubing to Stoppers and Rubber Tubing	8
2. The Set Up	8

	PAGE
XI. Collecting Gases.	8
1. By Displacement of Water	8
2. By Displacement of Air.	8
XII. Tasting and Smelling.	8
XIII. Weighing.	9
XIV. Determining Volumes	9

PART I.—GENERAL INORGANIC CHEMISTRY EXERCISES

1. Study of the Gas Burner.	12
2. Elementary Methods of Manipulating Glass Tubing.	12
3. Simple Metric Units.	13
4. Study of Properties	14
5. Study of the Composition of Various Substances	15
6. Study of Homogeneous and Heterogeneous Solid Systems	16
7. Study of Homogeneous and Heterogeneous Liquid Systems	17
8. Study of Phenomena Accompanying Chemical Change.	18
9. Study of the Replacement Series	19
10. Chemical Change Produced by Means of an Electrical Current	21
11. Study of the Experimental Method for Obtaining the Electro-chemical Series.	22
12. Study of the Methods of Chemical Change.	23
13. The Analytical Balance	24
14. Determination of Percentage by Volume of Oxygen in Air.	25
15. Preparation of Oxygen and Formation of Oxides	27
16. Preparation of Hydrogen and Its Use as a Reducing Agent	28
17. Reduction of Ferric Chloride.	29
18. Oxidation of Ferrous Sulphate	30
19. Water as a Solvent and Reagent	31
20. Study of a Natural Water	31
21. Study of Some Acids and Acidic Oxides	32
22. Study of Some Bases and Basic Oxides.	33
23. Neutralization and the Formation of Salts	34
24. Preparation and Properties of Hydrogen Peroxide.	36
25. Determination of Chemical Equivalent by Replacement.	37
26. Determination of Chemical Equivalent by the Eudiometer Method	38
27. Preparation and Properties of Chlorine	41
28. Preparation and Properties of Hydrochloric Acid	42
29. The Chlorides of the Alkali Group	43
30. The Chlorides of the Alkaline Earth Group.	44
31. The Chlorides of the Aluminum Group	44
32. The Chlorides of the Zinc Group	45
33. The Chlorides of the Arsenic Group.	45
34. The Chlorides of the Copper Group.	46

CONTENTS

ix

	PAGE
35. The Chlorides of the Silver Group.	47
36. Bromine and Iodine.	48
37. Preparation and Properties of Hydrogen Sulphide.	49
38. Solubility of Sulphides in Acid and Alkaline Solutions.	50
39. Preparation and Properties of Ammonia.	52
40. Systems, Colloidal Solutions and Dialysis	53
41. Solubility of Gases, Liquids and Solids in Water	54
42. Elevation of the Boiling Point	54
43. Depression of the Freezing Point	55
44. Osmosis and Osmotic Pressure	55
45. Relative Conductance of Solutions	56
46. Concentration of Solutions—Acidimetry and Alkalimetry	57
47. Oxycompounds of Chlorine and their Reactions.	58
48. Preparation of a Hard Bleach Solution.	58
49. Preparation of Soft Bleach Solution.	59
50. Properties of Chlorates and Perchlorates.	59
51. Properties of Bromine and of Iodine Compounds	60
52. Preparation and Properties of SO_2	61
53. Properties of Sulphurous Acid	61
54. Preparation and Properties of Sulphuric Acid.	62
55. Tests for Sulphur in Compounds	63
56. Sulphates	63
57. Preparation of a Plaster of Paris Cast.	64
58. Properties of Aluminum Sulphates and Alums	64
59. Pyrosulphates and Persulphates.	65
60. Thioacids and Thiosulphates.	65
61. Nitric Oxide and Nitrogen Dioxide	66
62. Preparation and Properties of Nitric Acid	66
63. Nitrous Oxide	67
64. Nitrous Acid and its Salts	67
65. Orthophosphoric Acid and Orthophosphates	68
66. Sodium Metaphosphate Bead.	68
67. Amphoteric Character of Metallic Elements	69
68. Properties of Manganese Dioxide	71
69. Preparation of Manganates by Fusion.	71
70. Test for Manganese in Compounds	72
71. Preparation of Chromium Trioxide	73
72. Properties of Chromium Compounds	73
73. Preparation of Chromates and Their Reduction	73

PART II—ORGANIC CHEMISTRY EXERCISES

74. Preparation of Methane	76
75. Preparation of Ethylene.	76

	PAGE
76. Preparation of Acetylene.	77
77. Preparation of Gasoline and of Kerosene.	77
78. Preparation of and Tests for Methanol, Carbinol or Methyl Alcohol	79
79. Preparation of and Tests for Ethanol, Methyl, Carbinol or Ethyl Alcohol	79
80. Properties of Glycerol.	80
81. Molisch Reaction for Carbohydrates.	80
82. Some Properties and Tests for Different Sugars.	80
83. Formation of Caramel.	81
84. Inversion of Sucrose.	81
85. Determination of the Presence of Reducing Sugar in Milk.	82
86. Preparation of Starch Paste and Tests for Starch	82
87. Conversion of Starch to Reducing Sugars	82
88. Solubility of Cellulose and Schweitzer's Reagent	83
89. Preparation of Parchment Paper	83
90. Analysis of Textile Fabrics for Wool, Cotton and Silk.	83
91. Preparation of Acetic Acid.	85
92. Analysis of Vinegar	85
93. Preparation of Soap.	85
94. Analysis of Soap	86

PART III—QUALITATIVE ANALYSIS

I. Basic Constituents	91
A. Preliminary Classification into Groups.	91
Graphic Method of Representing the Procedure.	92
B. Routine Qualitative Procedure for the Basic Constituents	93
Group I Procedure	93
Group II Procedure	94
Group III Procedure	97
Group IV Procedure	99
Group V Procedure	100
C. Equations Indicating the Reactions Occurring in the Procedure	101
Group I Reactions.	101
Group II Reactions.	102
Group III Reactions.	106
Group IV Reactions.	109
Group V Reactions.	110
II. Acidic Constituents.	110
A. Preliminary Examination of Solid Unknown Systems	111
B. Systematic Examination of Unknown System for Acidic Constituents	112

CONTENTS

xi

	PAGE
C. Special Tests for Acidic Constituents.	113
Division I. Chlorides, Bromides, Iodides.	114
Division II. Sulphates, Carbonates, Sulphites, Thio- sulphates, Borates, Phosphate	114
Division III. Nitrate	115
III. Preparation of the System for Analysis	115
A. Insoluble Substances.	116
B. Preparation of the Solution for Analysis	117

APPENDIX

Flame Colorations and Bead Tests	120
Methods of Preparations of Reagents.	122
INDEX.	125

LIST OF ILLUSTRATIONS

	PAGE
Figure 1. Correct and Incorrect Methods of Bending Glass Tubing	4
Figure 2. Apparatus for Distilling	6
Figure 3. Wash Bottle	13
Figure 4. Balance Scale.	25
Figure 5. Simple Hemple Burette and Leveling Tube.	26
Figure 6. Absorption Pipette.	27
Figure 7. Apparatus for Preparation of Oxygen.	27
Figure 8. Apparatus for Preparing Hydrogen and Showing its Property as a Reducing Agent.	29
Figure 9. Apparatus for Determining Chemical Equivalent by the Eudiometer Method. Klug's Design.	39
Figure 10. Apparatus for Determining Chemical Equivalent by the Eudiometer Method. Exner's Modification	39
Figure 11. Apparatus for Determining Chemical Equivalent by the Eudiometer Method. Egloff's Design	40
Figure 12. Apparatus for the Preparation of Chlorine	41
Figure 13. Apparatus to Show the Electrical Conductance of Solutions.	56
Figure 14. Burettes and Burette Holder	57

LABORATORY MANUAL IN GENERAL CHEMISTRY

INTRODUCTION

Picture to yourself an Indian scout, in the early days of the exploration of our country, as he picks his way through the dense woods searching for evidence of unfriendly Indians. You picture him as being alert in every way. He is looking, listening, smelling, feeling—employing every sense in every way to catch the slightest sign. How adeptly he uses his hands to spread noiselessly the brush that blocks his vision! How accurately he gauges distances and directions! How clean and neat he keeps his gun so that it is sure to work at an instant's notice! How sure he is that his powder is dry and that his knife is sharp! How careful he is to check all his observations that his report may later be accurate and complete! He knows that the success of his mission and, perhaps, his very life depend upon constant attention to details. He knows that he must depend upon himself. His brain is keenly active, directing him constantly and enabling him to keep a mental control over his whole plan of action.

How like such an Indian scout is a person while performing an experiment in the laboratory. He is literally a scout, picking his way in search of evidence of chemical truths. Constantly alert must he be for the slightest sign. He must use his hands continually to build and operate apparatus to help his observation. Dimensions and directions must be accurately gauged. He must keep his apparatus clean and his chemicals pure. He must check his observations and be sure his notes are correct and complete. The success of his work and sometimes his safety depend upon his attention to details. He should depend upon himself. His brain must be keenly active, and he must keep a mental control over the complete experiment.

To become a good experimenter, a person might well study a Boy Scout manual. In the chemical laboratory, a person should aim (1) to learn to construct and handle apparatus skillfully (manipulate); (2) to cultivate habits of observation; (3) to become intimately acquainted with many pure substances, solutions, and mixtures; and (4) to learn to reason about data obtained by observation. This will result in the development of the scientific method of thought and work.

A laboratory worker is constantly asking questions of nature. Each experiment is a question in natural language. The answer comes back in the same language, sometimes with compelling forcefulness, and sometimes in accents so low that it is difficult to catch, but always an absolutely truthful answer to the question as asked. The question must be asked clearly, if a clear answer is to be obtained. If the experiment is performed properly, the experimenter will be able to draw a conclusion that he can label confidently with the familiar **Ans.** This label at the end of any problem should indicate from their initial letters, *Accuracy*, *Neatness* and *Sureness*. Cultivate habits that arise from following the directions given in the next paragraph, if you would use the label, **Ans.**, to mean what it should.

Laboratory Directions.—1. Study the entire experiment with the aid of the textbook before coming to the laboratory. Based upon this study, plan your work for the entire experiment before starting. Ask an instructor about points not clearly understood. “Plan your work and work your plan. Always remember that you will get no more out of your work than you put into it.”

2. Bring your textbook to the laboratory each day.

3. Work independently of other students, unless instructed differently.

4. Arrange your apparatus neatly, away from the edge of the desk, so that it will not be upset. You will then have plenty of room in which to work.

5. Do not look directly into a vessel containing matter being heated.

6. Always clean apparatus before putting it away, so that it will be available for use instantly.

7. Always clean the desk top and reagent bottles before leaving the laboratory at the end of the period.

8. Report immediately all accidents, cuts, or burns to an instructor.

Manipulation

Your hands are wonderful tools with which you are constantly equipped. **Use your hands.** To use your hands effectively in the laboratory, you can well learn from the experience of others. Of the many ways of doing a thing, a student should try to learn the best. Study the following directions for doing particular operations, and practice them at every opportunity until they become habitual. These directions are grouped together here for ready reference.

I. Pouring Liquids from Containers.—Do not place the stopper of the bottle on the desk so that the part that enters the bottle comes in contact with the desk. It is better, however, to hold the stopper between the fingers, letting it project from the back of the hand. Remove the drop which usually sticks to the mouth of the bottle, by touching it to the top of the receiving vessel, the stopper, or a stirring rod.

In pouring liquids from a beaker, moisten a glass rod with the liquid, hold it firmly against the edge of the lip of the beaker, and pour the liquid down the rod. Always prevent the last drops from running down the outside of the vessel out of which it is poured, by removing them with the stirring rod.

II. Transferring Dry Solids.—In transferring dry solids from a bottle, rotate the bottle and collect the solid on a small piece of paper. By means of this, introduce the solid into the test tube or receiving vessel. To avoid getting the material on the sides of a test tube, fold a narrow strip of paper into a V-shaped trough which will slip into the tube; place the material in this, tip the tube to a horizontal position, and insert the paper into the tube. This will permit the material to slide off the paper onto the bottom of the vessel when it is brought into a vertical position.

III. Manipulating Glass Tubing.

(a) *To Cut Glass Tubing.*—Scratch with a file, the place where the glass tubing is to be cut. Remember that a file is not a saw; push the file across the tubing and do not draw it back and forth. After a distinct mark has been made on the tubing, grasp it firmly in both hands, placing the thumbs back of the mark. Push the thumbs against the tubing and pull with the hands at the same time. The tube will break at the desired place.

(b) *To Fire Polish Ends of Glass Tubing.*—Hold the end of the tube in the flame and rotate just long enough to melt the sharp edge. Do not permit the inside diameter to decrease appreciably.

(c) *To Bend Glass Tubing.*—The fish-tail or wing-top is to be used on the burner when bending glass tubing. It gives a wide, thin flame which should be so regulated as to be of uniform temperature throughout, in order not to soften the glass more at one part than another. Place the glass tubing lengthwise in the flattened flame. The tubing should be kept in constant motion by rotating and moving it slightly from side to side. When it is hot enough so that it begins to soften and bend, remove it from the flame, hold it firmly, and bend it into shape (**Fig. 1**). Retain it in this position until the glass hardens. The ends should always be pulled upward, thus permitting the glass tubing to sag downward.

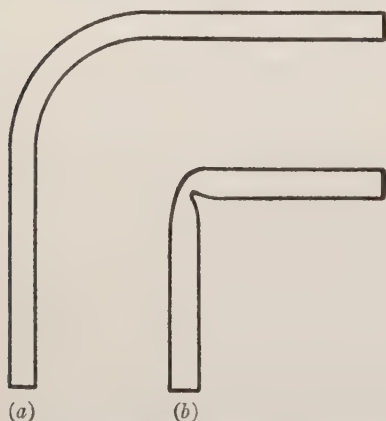


Fig. 1.—(a) This Shows a Correctly Made Right-angle Bend. (b) This Shows a Common Result of Incorrect Heating and Bending.

It is always well to *anneal* the tubing after bending so as to make it less brittle. This is done by holding it in the yellow flame of the burner until soot is deposited upon it. When it is cool, wipe off the soot with a piece of paper or cloth.

(d) *To Draw or Seal a Glass Tube.*—Introduce the tube into the flame and rotate it until it becomes soft. Remove from the flame and draw out slowly until it is as thin as desired. Hold this in position until it hardens. If it is desired to seal the tube,

cut at the constricted portion as described above. Then hold the constricted end in the flame, rotating until the edges melt together and the end is closed. By reheating, and blowing into the tube, the end may be blown out and a rounded end produced. By heating the tube and pulling it quickly, it may be pulled apart and the separate ends rounded.

IV. Heating.—All laboratory vessels should be dry on the outside before heating and, at first, heated gently.

Never look directly into an open vessel that is being heated. In general, never hold your face near any vessel that is being heated.

Glass test tubes containing a liquid may be heated in the free flame. They may be held in the fingers, by a test-tube holder, or

by a strip of paper folded in the form of a band. Avoid heating them directly on the bottom, when they contain heavy liquids or liquids in which there is a precipitate. Heat the upper part of the liquid first. Gently shake the test tube with a rotary motion while heating.

Beakers and flasks containing liquids should be placed on a wire gauze or an asbestos mat when it is necessary to heat them. Care should be taken that the flame does not lick up the side of a beaker and unduly heat the glass above the liquid. This is very apt to break the beaker. Bottles and watch glasses should not be heated with a free flame.

Porcelain crucibles are usually supported by a triangle, and heated in the free flame. Do not heat evaporating dishes in the free flame, but place them on a metal gauze or an asbestos mat. If, in evaporating a solution, the dish becomes dry, allow it to cool before adding any liquid.

Sometimes it is desirable to heat a system at a low or a constant temperature. Among the different methods used, the following are common:

1. *Water Bath*.—The vessel containing the system to be heated is partially immersed in water, which in turn is heated. This applies the heat uniformly over a surface of the system at a temperature the same as that of the water. The temperature cannot rise above the boiling temperature of the water. Often a test tube is immersed in water in a beaker, which is thus employed as a water bath.

2. *Steam Bath*.—The vessel containing the system to be heated is partially immersed in steam. This applies the heat uniformly over a surface of the system at the temperature of the steam. A watch glass placed on the top of a beaker containing boiling water is used to evaporate small amounts of liquids.

3. *Sand Bath*.—The vessel containing the system to be heated is partially buried in sand. This applies the heat fairly uniformly and slowly.

4. *Air Bath*.—The vessel containing the system to be heated is supported at only a few points in a vessel containing air which can be heated to the temperature desired. This applies the heat very uniformly.

5. *Oil and Molten Salts*.—These may be used in a manner similar to water in a water bath, when the temperature at which the heating is to be done is higher than that of boiling water.

6. *Hot Plate*.—This is a metal plate (which may be covered by a sheet of asbestos) heated from beneath by gas, electricity, oil, coal, or any other primary source of heat. This is very useful in heating inflammable liquids.

V. *Evaporating*.—To secure rapid evaporation, the system should: (1) be contained in a vessel shaped to expose the maximum surface of the system to the atmosphere; (2) be heated to its boiling temperature; and (3) have the atmosphere near its exposed surface constantly swept clear of the vapor being formed. Evaporating dishes are shaped purposely to expose a large surface of the contained liquid. Spattering of a liquid is to be carefully avoided.

VI. *Distilling*.—The separation of the more volatile part of a system from the less volatile part may be effected by distillation (Fig. 2). Distilled water used in the laboratory is the condensed volatile portion of ordinary tap water.

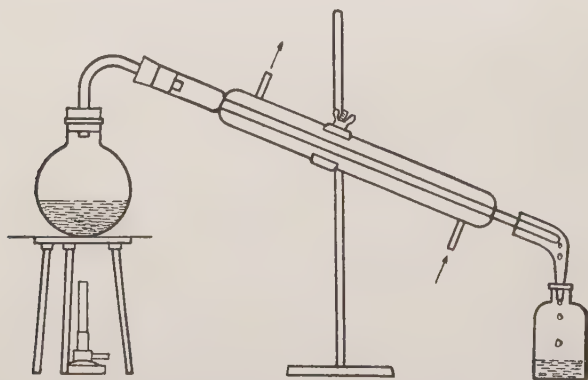


Fig. 2.—Apparatus for Distilling. The Arrows Show the Direction of Flow of The Condensing Water.

VII. Separating a Solid from a Liquid.—

1. *Decantation*.—After a solid dispersed throughout a liquid settles, the liquid may be separated from the solid by simply pouring it off carefully into another vessel, without disturbing the solid. This process is called decantation.

2. *Filtration*.—The separation of a solid from a liquid is usually accomplished by the process of filtration. The precipitate is retained on the filter paper and the liquid passes through. This liquid is termed the filtrate.

(a) *Folding the Filter Paper*.—Fold a circular disc of filter paper to form a semicircle, and fold this again, but do not bring

the two edges quite together, thus making one segment slightly larger than the other. Open the larger segment, and press the filter paper into the funnel. As the funnels do not always have a 60° angle, the larger segment usually fits the funnel. The upper edge of the filter paper should fit snugly against the funnel. Hold in place, and moisten with water to keep the paper in position.

(b) *Introducing the Liquid onto a Filter Paper.*—Pour the liquid down a moistened stirring rod, which nearly touches the paper, and directly upon the paper. Have the tip of the funnel so arranged that the liquid drops onto or near the side of the beaker, thus preventing spattering.

(c) *Removal of the Precipitate from a Beaker to a Filter Paper.*—Drain the liquid from the precipitate, and by means of a jet of water from the wash bottle, wash the precipitate from the sides of the beaker to the bottom. Now, tip the beaker gradually and pour the liquid and precipitate onto the filter. While the beaker is in this inverted position, flush the precipitate out with a jet of water from your wash bottle, thus transferring all of the precipitate to the filter paper. Use stirring rod as directed in I on page 3.

VIII. Washing Precipitates.—

1. *On Filter Paper.*—Allow the liquid to drain from the precipitate. Then pour the wash water around the upper edge of the filter paper, thus washing down the impurities which were in the mother liquid. Allow all of this to drain out of the funnel. Now wash again by pouring the wash water onto the upper edge of the filter paper and allow this to run through. Continue the washing by always adding the wash water to the upper edge of the filter paper until the precipitate is thoroughly washed.

2. *By Decantation.*—When the liquid has been carefully poured through the filter paper without disturbing the precipitate remaining in the original beaker, a quantity of the washing liquid is poured upon it and stirred thoroughly. The precipitate is allowed to settle, and the liquid is decanted through the filter paper. This process may be repeated until the precipitate is thoroughly washed. This method is especially valuable in washing jelly-like precipitates.

IX. *Boring Stoppers.*—A cork borer of proper size should be selected and sharpened if necessary. The borer should be pressed firmly against the cork and, by a rotary motion, forced into the cork, thus cutting a hole through it. When the borer is

nearly through, place the end of the cork against a soft surface such as a piece of linoleum or pine and, by rotation, work the borer through the cork. The cork may be bored from both ends. To bore a rubber stopper, moisten the borer with water or sodium hydroxide solution.

X. Constructing Apparatus.—All apparatus should be carefully constructed so that it presents a neat appearance. One of the most important values of laboratory work is the skill acquired. This can be best acquired by paying special attention to the detailed construction of apparatus. The following are some of the essential points:

1. *Fitting Glass Tubing to Stoppers and Rubber Tubing.*—Always see that the ends of glass tubing have been rounded off by fire polishing. Never attempt to introduce a piece of glass with sharp edges into a piece of rubber tubing or a stopper. It is always well to wet the glass tubing with water and to insert it by means of a rotary motion.

2. *The Set Up.*—Always have the bends of glass tubing well made and bent at the correct angles to give the apparatus a neat appearance. Have a reason for everything in the set up.

The tubing for the exit of gases should protrude *just below* the under surface of the cork. Insert corks into flasks with a rotary motion and remove them in the same way.

When clamping apparatus, fasten the clamps just tight enough to hold the apparatus in place, but have everything secure and firm.

Always have your apparatus clean. Remember that soap and water are very effective for this purpose.

XI. Collecting Gases.—Depending upon their properties, gases may be collected by displacement of water or of air.

1. *By displacement of water.*—This consists in filling a vessel with water and then inverting this in a trough and permitting the gas to pass into the vessel, thus replacing the water.

2. *By displacement of air.*—This consists in allowing the gas to flow from the delivery tube whose mouth is as far from the mouth of the containing vessel as possible, thereby forcing the air out. If the gas is heavier than air, the air is forced upward; if the gas is lighter than air, the air is forced downward. The bottles of gases are covered with glass plates until needed.

XII. Tasting and Smelling.—The general principle of not tasting the substances in the laboratory nor of eating while in

the laboratory should be adopted. Never taste any of the materials, unless specifically directed to do so.

In determining the odor of materials, and particularly of gaseous substances, do not place your face directly over the container. By means of your hand, waft the fumes toward you, and thus obtain sufficient of the material to ascertain its odor.

XIII. Weighing.—The weight of a body is determined by means of a balance. If directed to use a balance, make sure you understand its use thoroughly before starting. Ask an instructor to explain the balance to you.

XIV. Determining Volumes.—The volumes of liquids and gases are measured by narrow tubes known as graduated cylinders and burettes. The top surface of the liquid in a tube of small diameter is usually curved. This curved surface is called the **meniscus**. The reading on the scale is made in a horizontal plane tangent to the meniscus.

PART I

GENERAL INORGANIC CHEMISTRY EXERCISES

Exercise No. 1

STUDY OF THE GAS BURNER (313-315)¹

(a) Examine the gas burner carefully. Take it apart and study its construction. See that all parts are clean, and that the rubber tubing is in good condition.

(b) Follow through the passageway for the gas supply and the port holes for the air supply. Close the air ports and light the gas. Regulate the quantity of gas entering the burner so that the height of the flame is 10 centimeters. Notice the appearance of the flame. Slowly increase the amount of air and observe the changes in the appearance of the flame.

(c) The non-luminous flame is used for practically all laboratory operations requiring heat. This is best obtained by starting with the yellow flame and admitting air until the yellow flame just disappears. The burner should operate quietly. A noisy burner indicates an excess of air. The yellow flame is used in conjunction with the blowpipe, and for operations in which a deposit of soot is required.

Exercise No. 2

ELEMENTARY METHODS OF MANIPULATING GLASS TUBING

(a) Study carefully the method of cutting glass tubing under (IIIa), page (3). Cut three pieces of tubing, each 4 inches in length.

(b) Fire-polish the ends of the pieces cut in (a) according to the method given under (IIIb), page (3).

(c) After you feel that you understand the method for bending glass tubing as given under (IIIc), page (4), practice making a 90° bend, an obtuse-angled bend and an acute-angled bend. Samples of these bends are displayed for your benefit.

¹ Numbers in parentheses indicate pages of the authors' "General Chemistry" to which the student is referred.

(d) Glass tubing may be drawn out by the method described under (III d), page (4). Practice this operation a few times. Cut one piece of tubing about 7 inches long. Heat a short length at the middle uniformly and pull apart quickly. Then, round the ends in the manner described under (III d), page (4). Save the two tubes made for use in subsequent exercises.

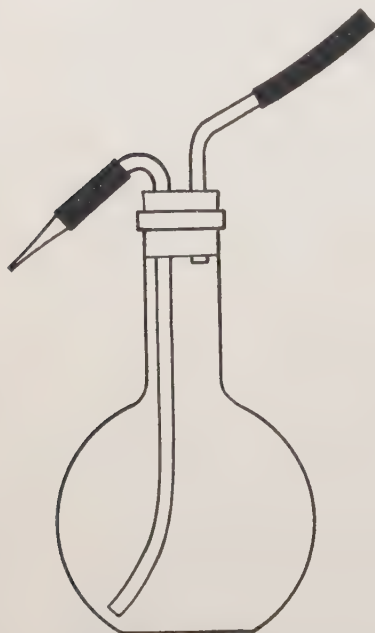


Fig. 3.—Wash Bottle.

(e) When you feel that you have acquired a reasonable degree of proficiency in the above operations, prepare a wash bottle as illustrated in Fig. 3. Have the wash bottle inspected and accepted by the instructor, and keep it for future use.

Exercise No. 3

SIMPLE METRIC UNITS (8-9)

(a) Using a metric scale, draw two parallel lines one millimeter apart. Draw squares whose sides are 1 centimeter, 5.6 centimeters, 67 millimeters, 1 decimeter, and 1 inch. Note the relative sizes of a square centimeter and a square inch.

How many square centimeters in a square inch? How many cubic centimeters in a cubic inch?

(b) Measure the length of the upright rod on your ring stand, and express your measurement both in centimeters and in inches.

(c) What is the mass in grams of your dry graduated cylinder? When the cylinder is balanced on the scales, add a 50 g weight to the other pan, and then pour water into the cylinder until the masses on the two pans again balance. What is the mass in grams of water added? What is the volume in milliliters of the water added? What is the volume in milliliters of 1 gram of the water?

(d) Using the graduated cylinder, measure accurately into a clean, dry test tube 5 ml of water. Make a file mark on the test tube opposite the bottom of the meniscus. Add 5 ml more, and

mark as before. Try to remember the positions of the 5 and 10 ml marks on the test tube.

(e) Pour into each of your beakers and the Erlenmeyer flask a volume of water as marked upon them. Note carefully the level of the top surface of the water in each when each is filled to its designated capacity.

(f) Estimate the amounts of water that occupy respectively 25 ml, 50 ml, and 100 ml. Test the accuracy of your estimation by pouring the water into a graduated cylinder.

Exercise No. 4

STUDY OF PROPERTIES (1-2)

A system is an isolated portion of matter set apart to be studied and considered. Systems are described and distinguished by their characteristics which are termed **properties**.

Some properties readily observed comprise: the physical states of matter (solid, liquid, and gas), color, odor, taste, conductivity of heat and electricity, and solubility in various solvents.

A varied list of substances that may be studied contains: chalk, copper, copper sulphate, sugar, salt, carbon, lead nitrate, marble, sulphur, starch, and aluminum. Study the substances designated by the instructor from the above list with respect to their state of matter, color, and solubility in water.

(a) **State of Matter**.—This is recorded as solid, liquid, or gas.

(b) **Color**.—In noting color, distinguish between the terms, **colorless** and **white**. The glass of the test tube is colorless, but when the tube is ground to a fine powder it is white.

(c) **Solubility in Water**.—Transfer about 5 ml (5 to 8 g) of the designated system to a small beaker, and add 20 ml of water. Place the beaker on the wire gauze, and heat gently for 5 minutes. While the beaker is being heated, arrange the funnel and filter stand according to directions given under **VII** on Filtration, page 6. When the heating is concluded, transfer the contents of the beaker to the filter paper, following carefully the directions given in (a), (b), and (c) under **VII**. In general, when filtering, do not have the liquid on the filter higher than $\frac{1}{4}$ inch below the rim of the paper. As the liquid filters through, add more from the beaker to the paper until all has been filtered. Be sure to place a beaker under the funnel before beginning the filtration.

While the liquid is filtering, set up a small steam bath as follows: A beaker containing 50 ml of water is covered with the watch glass and placed on the wire gauze over the burner. The liquid to be evaporated is placed on the watch glass. A watch glass is never heated in any other way.

When the filtration is concluded, evaporate the filtrate to dryness by adding it in small portions to the watch glass. Compare the amount of solid left on the watch glass with the amount on the filter paper or left in the beaker. **From this comparison,** draw your conclusion as to the solubility of the system.

Note.—Start the determination of solubility first, and use the time during which evaporation is in progress for other work.

Present the results in tabular form as follows:

TABLE OF PROPERTIES

<i>System</i>	<i>State of Matter</i>	<i>Color</i>	<i>Solubility in Water</i>	<i>Remarks</i>
---------------	------------------------	--------------	----------------------------	----------------

Note.—In expressing solubility, use only the following terms: “very soluble,” “moderately soluble,” “slightly soluble,” and “insoluble.”

Exercise No. 5

STUDY OF COMPOSITION OF VARIOUS SUBSTANCES (3-4)

(a) **Composition of Mercuric Oxide.**—Introduce mercuric oxide into an ignition tube (Exercise No. 2) to a depth of about 5 mm. Heat the sealed end gently until all the mercuric oxide has disappeared. During the heating, hold a glowing splint just inside the mouth of the tube. The splint glows brilliantly and may burst into flame. This is a test for oxygen. Bright globules of mercury may be observed on the side of the tube, if it has not been heated too much. The mercuric oxide has been **decomposed** into gaseous oxygen and liquid mercury. This illustrates **analysis**. Transfer the residue of the materials in this experiment to wide-mouth bottles provided for the purpose. Tabulate the properties of the original and final substances, and justify the statement that this is analysis.

(b) **Composition of Magnesium Oxide.**—Hold a piece of magnesium ribbon by the tongs or forceps just above a porcelain crucible. Ignite the magnesium in the burner flame. Observe the brilliant light and the white substance which has fallen into

the crucible. The latter is magnesium oxide and has been **synthesized** from the oxygen of the air and magnesium. Tabulate the properties of the original and final substances, and justify the statement that this is synthesis.

(c) **Composition of Zinc Sulphide.**—Mix 1 ml (2 g) of powdered zinc and 3 ml (3 g) of powdered sulphur by grinding in a mortar. Place on an asbestos mat, or piece of asbestos paper, in the hood. Hold the flame of the burner in contact with the mixture until the latter begins to glow. The reaction continues vigorously after the flame is removed. The substance formed is zinc sulphide, synthesized from zinc and sulphur. Tabulate, and justify the statement that this is **synthesis**.

Exercise No. 6

STUDY OF HOMOGENEOUS AND HETEROGENEOUS SOLID SYSTEMS (4-7)

If a system can be separated by physical means into parts which differ in their properties, the system is **heterogeneous**. **A system is homogeneous or heterogeneous with respect to the method which is employed to test for homogeneity or heterogeneity.** Due to new methods of testing, and the refinement of old methods, many systems previously thought to be homogeneous have been found to be heterogeneous. Three generally used methods are (1) the unaided eye, (2) the use of the microscope, (3) the use of solvents.

Study the systems designated as **A, B, C**, and so forth by the instructor. A varied list contains (1) salt-chalk, (2) copper sulphate-carbon, (3) sand-salt, (4) litharge-naphthalene, (5) ferric oxide-sodium carbonate, (6) salt, (7) chalk, (8) copper oxide, (9) sodium chloride—benzoic acid, (10) ferric oxide-benzoic acid.

Take about 4 ml (5 g) of the system for study.

(a) Study the system with the unaided eye. Are all the particles alike in color? The size of a particle is not a property; hence, differences in the dimensions of the particles are not to be taken as evidence of heterogeneity.

(b) Place some of the system upon a small watch glass, and examine under a low-power microscope. What differences other than size do you observe in the appearances of the particles?

(c) Study the solubility of the system in water, by the method given under (c) Exercise No. 4. If the system is completely soluble in a given solvent, it is homogeneous with respect to that solvent. **Compare the physical and chemical properties of the residue upon the filter paper with the corresponding properties of the residue obtained by evaporation upon the watch glass.** If the two residues differ in their properties, the system is heterogeneous. The use of solvents as an aid in the classification of systems presupposes that no chemical reaction occurs between the solvent and the system.

(d) If the system is found to be homogeneous with respect to the unaided eye, the microscope, and the solvent, water, study its solubility in benzene. Add to the system, in a 50 ml beaker, 10 ml of benzene. Stir for about 3 minutes, then filter through a dry filter paper. Do not heat, because the solvent is **inflammable**. Have your water bath boiling, turn off the gas, and evaporate the benzene filtrate by pouring small portions on the watch glass.

Tabulate the observations taken under (a), (b), (c), and (d), and **justify** your conclusion as to whether the system is homogeneous or heterogeneous.

Exercise No. 7

STUDY OF HOMOGENEOUS AND HETEROGENEOUS LIQUID SYSTEMS (6-7)

In the classification of liquid systems, the unaided eye and the microscope are the methods usually employed. Study the systems designated by the instructor. A varied list includes (1) water-acetone, (2) water-benzene, (3) acetone-benzene, (4) acetone-carbon disulphide, (5) water-chloroform, (6) alcohol-gasoline, (7) carbon tetrachloride-water.

(a) The above designated systems will be prepared and studied in the following manner: Measure 5 ml of the first-mentioned liquid, transfer to a test tube, and add an equal volume of the second liquid. Shake *vigorously* and observe whether the two liquids form a homogeneous system or separate into two distinct layers. It is unnecessary to use the graduated cylinder each time, if the volume occupied by 5 ml of water be noted, and the same size test tube be used. All test tubes must be clean and **dry**. Tabulate your observations.

(b) Examine a sample of milk with the unaided eye, and then examine the specimen under a high-power microscope, the latter being arranged by the instructor. Classify the system, milk.

(c) Add 5 ml (5–7 g) of cane sugar to 25 ml of water. Heat for a moment, at the same time stirring. Is the resulting system homogeneous or heterogeneous? Again add the same amount of sugar to the system with heating and stirring. Does the system change? Add a third portion of sugar to the system. Is there a change in the homogeneity or heterogeneity of the system? What is the definition of a solution?

(d) Shake 2 ml (2–3 g) of precipitated chalk with 10 ml of water in a test tube. Heat for a moment. Does the chalk dissolve? Is the system homogeneous or heterogeneous? Justify your answer.

Exercise No. 8

STUDY OF PHENOMENA ACCOMPANYING CHEMICAL CHANGE (7-8)

From now on, use chemical equations as much as possible in the representation of chemical changes.

(a) **Generation of Heat.**—Pour 5 ml of concentrated hydrochloric acid into 5 ml of concentrated sodium hydroxide solution in a small beaker. Stir gently, and observe the heat which is generated. Observe the heat which is generated when the oxygen of the air combines with the gas from your burner.

(b) **Generation of Light.**—Ignite a piece of magnesium ribbon. Observe the brilliant light.

(c) **Generation of Electrical Current.**—The apparatus for this consists of two electrodes of different metals, immersed in a solution of some acid. The two electrodes are connected to the terminals of an instrument used to indicate the presence of an electrical current. A convenient arrangement consists of rods of copper and zinc dipping into dilute sulphuric acid and connected to a galvanometer. Sketch the apparatus arranged by the instructor. Immerse the zinc rod in the acid, and read the galvanometer. Remove the zinc rod, and wash with water. The copper rod is left permanently in place. The zinc and acid react and, by this particular device, produce an electrical current. In the process, zinc sulphate is formed and remains dissolved.

(d) **Generation of Gas.**—Make a solution of sodium carbonate by adding 2 ml (2–3 g) to 10 ml of water in a small beaker. Shake

until dissolved. Add 2 ml of dilute sulphuric acid, drop by drop. What do you observe?

Treat a small piece of copper with 1 ml of concentrated nitric acid. What do you observe?

In boiling a solution, be careful to distinguish between bubbles of steam due to boiling only, and gas which is liberated in a chemical reaction.

(e) **Change of Color.**—To 5 ml of dilute potassium dichromate solution, add 1 ml of hydrochloric acid and then 1 ml (1–2 g) of sodium bisulphite. Shake, and warm gently. Describe the color change. Treat 10 ml of potassium permanganate solution in the same manner. Describe the color change.

(f) **Formation of a Precipitate.**—If two clear solutions are mixed and a solid substance is formed, which may or may not settle to the bottom of the tube, precipitation has occurred. The solid substance is called the **precipitate**. Precipitates are described by their color and general appearance, the latter being denoted by the following terms: flocculent, curdy, gelatinous, amorphous, crystalline, finely divided, and non-settling. (Ask instructor for explanation of terms.) Other terms are used, but those given suffice for present purposes. Describe the color and appearance of the precipitates formed when 5 ml of the solution in the first column is treated with 1 ml of the solution in the second column.

1. Barium chloride.	Sodium sulphate.
2. Ferric chloride.	Ammonium hydroxide.
3. Lead nitrate.	Potassium chromate.
4. Copper sulphate.	Sodium carbonate.
5. Copper sulphate.	Potassium ferrocyanide.
6. Cobalt nitrate.	Sodium carbonate.
7. Calcium chloride (saturated).	Sodium carbonate (saturated).

Exercise No. 9

STUDY OF THE REPLACEMENT SERIES (14-16)

All metals used should have a fresh surface exposed by scraping or filing. Do not confuse the replacement of hydrogen with bubbles of steam due to boiling. When hydrogen is replaced, a continuous evolution of gas occurs, whereas the formation of bubbles of steam on a metallic surface ceases when the system is cooled. The *replaced metal* may appear in the following forms:

(1) finely divided gray or black particles; (2) curdy particles; (3) separate crystals; (4) a mat of interlaced crystals; (5) a burr or fuzz of crystals radiating from a point on the replacing metal; (6) loose scales or plates; (7) a smooth adherent layer upon the *replacing metal*.

(a) **Iron and Copper.**—Immerse a piece of iron in 5 ml of copper sulphate solution. Allow to stand until the end of the laboratory period. Record any changes observed. Write the equation. If replacement occurs, describe the color and form of the replaced metal. Is your result in agreement with the electrochemical series?

(b) **Zinc and Hydrogen.**—Place a piece of zinc in 5 ml of dilute hydrochloric acid. Write the equation. Record any changes taking place in the system. Explain your result as in (a).

(c) **Copper and Hydrogen.**—Place a piece of copper in 5 ml of dilute hydrochloric acid. Warm gently. Record your observations, and explain as in (a).

(d) **Zinc and Tin.**—Study the systems, zinc in stannous chloride solution and tin in zinc sulphate solution. Record any changes taking place in each system. If replacement occurs, write the equation. Which metal replaces the other from solutions of its salts?

(e) **Zinc and Lead.**—Refer to the electrochemical series, and predict whether zinc will replace lead or lead will replace zinc. Verify your prediction by studying the systems, lead in zinc nitrate solution and zinc in lead nitrate solution. Write the equation where replacement occurs. Describe the color and form of the replaced metal.

(f) **Mercury and Copper.**—Study the system, copper in mercuric nitrate solution. Write the equation and describe the color and form of the replaced metal. Allow about 30 minutes for the reaction to proceed; then remove the copper and rub it with a cloth. Why is mercury placed below copper in the electrochemical series?

(g) **Iron and Hydrogen.**—Study the system, iron in hydrochloric acid. Describe any changes taking place in the system. Write the equation. Should iron be above or below hydrogen in the electrochemical series?

(h) **Silver and Copper.**—Into 5 ml of silver nitrate solution (desk reagent) place a piece of copper wire or foil. Allow to stand. What happens? Write the equation.

21 CHEMICAL CHANGE PRODUCED BY ELECTRICAL CURRENT

(i) Arrange the above elements in a column such that any one will replace those below it. Compare your list with the electrochemical series.

Exercise No. 10

CHEMICAL CHANGE PRODUCED BY MEANS OF AN ELECTRICAL CURRENT (14-16)

The apparatus for this experiment consists of a number of electrolytic cells arranged in series. One cell is so constructed that any gas appearing on the electrodes may be removed from the cell and identified. The Hoffman apparatus is very suitable for this. The solutions to be electrolyzed will be designated by the instructor. A list of solutions includes (1) dilute acid or dilute alkali; (2) copper sulphate; (3) silver nitrate; (4) nickel ammonium sulphate; (5) zinc sulphate.

Direction of Current.—Remove the air from the tubes in which gas is to be collected and connect to the source of electricity. Allow the electrolysis to proceed until sufficient gas has been collected for identification. Remove and identify the gases. Oxygen may be identified as in Exercise No. 5. If the glowing splint is extinguished and a slight explosion is heard, the gas is hydrogen. Hydrogen collects on the **cathode**, that is, the electrode by which the current leaves the cell. Knowing which is **anode** and **cathode** in this cell, follow through the train, and determine which electrode is the anode and which the cathode in each cell.

Allow the gas in the tubes to escape if necessary, and proceed with the electrolysis until you can readily distinguish the deposits of metals on the electrodes. Ascertain on which electrodes the metals are deposited, and the relation of these to the electrode on which the hydrogen collects.

Draw a diagram of each of the electrolytic cells, showing the direction of the current, and name all the various parts. Name the substances deposited on each electrode. Tabulate your results.

SOLUTION ELECTROLYZED	NAME OF SUBSTANCE DEPOSITED ON		REMARKS
	<i>Cathode</i>	<i>Anode</i>	

When the experiment is concluded, empty the cells into the bottles provided. Wash the cells and electrodes and leave everything in place.

Exercise No. 11

STUDY OF THE EXPERIMENTAL METHOD FOR OBTAINING THE ELECTROCHEMICAL SERIES (16-17)

The apparatus for this experiment consists of a galvanometer attached to two metal electrodes, one of which is always copper immersed in copper sulphate solution. The other electrode is one of the assigned metals, such as silver, zinc, platinum, lead, cadmium, and iron.

All metals used must have a fresh surface exposed by filing or polishing. Remove the electrodes from the cell immediately after taking the galvanometer reading. Wash in water, and wipe dry.

(a) **Direction of Current in External Circuit.**¹—Connect the copper and zinc electrodes to the galvanometer, and immerse them in the solution of copper sulphate. Close the switch, and note the **magnitude** and **direction** of the galvanometer reading to the right or left, as the case may be. Interchange the electrode connections if the needle does not indicate a reading on the scale. It is assumed that, under ordinary conditions, the current produced flows from the copper to the zinc in the **external** circuit. Note carefully the direction of the current in the external circuit with relation to the direction of the galvanometer reading.

When the copper-zinc electrodes are used, the zinc replaces the copper in the copper sulphate, forming zinc sulphate and free copper, the latter depositing on the copper electrode. Accompanying this chemical change is a redistribution of energy, manifested by the flow of electricity in the system.

(b) Keep the copper electrode fixed in position, and replace the zinc by each of the assigned metals. Tabulate your observations.

ELECTRODES	DIRECTION OF CURRENT IN EXT. CIR.	GALVANOMETER READING	ANODE	CATHODE
------------	---	-------------------------	-------	---------

¹ Figure given in text, page 16.

(c) Prepare a table in which the assigned metals are arranged in the decreasing order of their galvanometer readings. Compare your order with that in the electrochemical series.

Exercise No. 12

STUDY OF THE METHODS OF CHEMICAL CHANGE

(7-8, 23-26)

Every chemical change is accompanied by changes in the properties of the system, together with an energy change.

(a) **Direct Union (synthesis).**—Mix on a piece of paper, 2 ml (2 g) of powdered sulphur and the same amount of fine iron filings. Place the mixture in a soft glass test tube, and heat. Watch the system, and when it begins to glow, remove it from the flame. Break the tube and examine the newly formed compound. Treat some of it with dilute sulphuric acid and observe the odor of the gas.

Make another mixture of iron and sulphur, using the same quantities of each substance. Treat some of this mixture with dilute acid. Compare the odor of the gas with that obtained by treating the newly formed compound with the same acid. Examine both the newly formed compound and the mixture under the low-power microscope. Classify each as homogeneous or heterogeneous. Write the equation representing the reaction between iron and sulphur. **What observations did you make which indicate that a chemical change takes place when iron and sulphur are heated together?**

(b) **Decomposition (analysis).**—Place about 2 ml (2 g) of copper nitrate in a test tube. Heat in the hottest part of the flame for 5 minutes. The copper nitrate is decomposed into three substances: copper oxide, which remains in the tube; nitrogen dioxide (NO_2), a brown gas; and oxygen, a colorless gas. Is the residue in the tube soluble in water? Is copper nitrate soluble in water? **What observations did you make which indicate that a chemical change takes place when copper nitrate is heated?**

(c) **Substitution (replacement).**—Place 2 ml (2-3 g) of cast iron drillings (or 5 iron brads) in 5 ml of copper sulphate solution. Let stand for one hour. Does the system change in color? What is the red coating on the iron? Write the equation. **What observations did you make which indicate that a chemical**

change takes place when copper sulphate in solution reacts with metallic iron?

(d) **Metathesis (exchange of parts).**—Add three drops of barium chloride solution to 5 ml of sodium sulphate solution. The precipitate is barium sulphate. Describe its color and appearance. Write the equation. The other compound formed remains in solution. **What is the evidence which indicates that a chemical change has taken place?**

Exercise No. 13

THE ANALYTICAL BALANCE¹

The analytical balance is one of the most important instruments used by scientists. It is a delicate instrument; hence, care is required for its operation and to maintain it in good condition. The understanding of the balance is useful in the discussion of weight relations in chemistry.

Before doing any work with the balance, have the instructor explain its operation. Note particularly the precautions necessary to prevent damaging the instrument. The most important of these are: (1) Never open the balance door without arresting the beam. (2) Never arrest the beam or pans when the pointer is more than one division from the center of the scale. Avoid rough handling or jarring of any kind. The most serious damage is the chipping of the agate knife-edges.

Note particularly (1) beam, (2) beam arrest, (3) pans, (4) pan supports, (5) rider carriage and rider, (6) leveling glass and leveling screws, (7) central and terminal knife edges.

Examine the box of weights. **Notice that there is a particular place for each weight. Whenever a weight is taken from the balance pan, it is restored at once to its proper place in the box. Weights are never handled with the fingers, but always with the pincers in the box.** Study the information on page 8 in the text.

(a) **Determination of Rest Point.**—The *rest point* is the point on the scale at which the pointer would come to rest if friction at the knife edges were eliminated. Level the balance

¹ This experiment is placed here owing to the small number of good balances usually available for use in General Chemistry. This enables the instruction in the use of the balance to be given to large classes before the subsequent experiments involving weighings are reached.

and lower the beam and pan supports. Place the rider on the end of the beam and remove the rider as soon as the beam begins to move as shown by the pointer. Allow the pointer to swing past the center of the scale two or three times, and then begin to take readings on the scale when the pointer is at the end of its swing. Take three readings on one side and two on the other side of "10." The following method shows how this may be calculated from the readings.

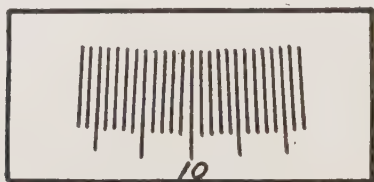


Fig. 4.—Balance Scale.

15.3	4.8	$\frac{45.2}{3} = 15.07$
15.1	5.1	
14.8	9.9	$\frac{9.9}{2} = 4.95$
45.2		
		$15.07 + 4.95 = 20.02$
		$\frac{20.02}{2} = 10.01$

(b) **Determination of the Weight of an Object.**—Place the object¹ on the left hand pan. Find a single weight in the box that is greater than the weight of the object, by placing the weight on the right hand pan, and very gently lowering the beam arrest until the pointer moves a short distance to the left. Replace the weight found with the next lower in the box. If this is found to be lighter than the object, leave it on the pan and then proceed in a **systematic order** down the box of weights. **Do not select weights at random.** Do not use the pan arrests until you begin to use the small weights. When you have gone through the smallest weight in the box, use the rider, and adjust its position on the beam so that the pointer swings equal distances on each side of the **rest point** which you calculated in (a). Add up the weights including that indicated by the position of the rider on the beam. What is the weight of the object given to you?

When you leave the balance, raise the beam and pan supports, close the balance door and be sure each weight is in its proper place.

Exercise No. 14

DETERMINATION OF PERCENTAGE BY VOLUME OF OXYGEN IN AIR (28-29)

The apparatus for this experiment consists of a measuring burette, leveling tube, and absorption pipette. The absorption

¹ This object may be the metal to be used in Exercise 26.

pipette contains metallic copper in 14 per cent ammonia solution saturated with ammonium chloride. This reagent is a good absorbent for oxygen gas.

(a) **Filling Burette and Leveling Tube with Water.**—Open the pinch-cock at the top of the burette (**Fig. 5**) and pour water into the leveling tube until both are about one-half full. Raise and lower the leveling tube three or four times until all trapped air has been removed from the rubber tubing. Finally, raise the leveling tube until the burette is completely filled. Close the pinch-cock. The surface of the water in the leveling tube should now be just visible. If not, adjust until satisfactory.

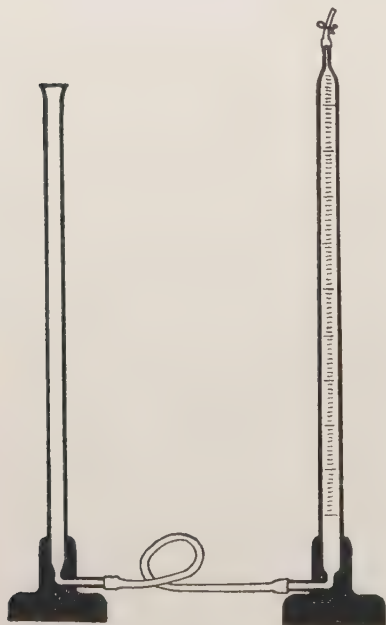


Fig. 5.—Simple Hempel Burette and Leveling Tube.

(b) **Taking the Sample of Air.**—Open the pinch-cock on the burette, raise the burette above the leveling tube until the former fills with air, taking care not to overflow the leveling tube. Close the pinch-cock. Hold the leveling tube against the burette and **keeping the surfaces of the water in the two tubes opposite each other, slowly open the pinch-cock until the two water surfaces are opposite the 100 ml mark**

on the burette. Close the pinch-cock. There is now 100 ml of air in the burette at atmospheric pressure.

(c) **Transference of Sample to Pipette.**—See that the pipette (**Fig. 6**) is full of liquid the surface of which is in the horizontal portion of the capillary tubing. Connect the burette to the pipette by U shaped capillary tube, leaving only enough room on the rubber tubing for the pinch-cocks to operate properly. Raise the leveling tube, and force the sample into the pipette taking care not to overflow the latter. Circulate the gas slowly back and forth by alternately raising and lowering the leveling tube

about 10 times, taking care that the liquid in the pipette is not drawn into the burette.

(d) **Reading Volume after Absorption.**—Lower the leveling tube, and draw the gas back into the burette. Close the pinch-cocks and disconnect the burette from the pipette. Place the leveling tube against the burette and with the surfaces of the water in the two tubes opposite each other, read the volume of the gas in the burette. This volume, subtracted from the volume of the sample, gives the volume of oxygen absorbed. Perform the analysis three times. Calculate the percentage of oxygen in air by volume, and report the average result.

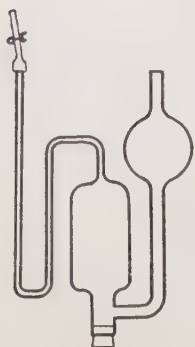


Fig. 6.—Absorption Pipette.

Note: Be sure the pinch-cock on the pipette is closed when you leave the apparatus; otherwise, the solution will continue to absorb oxygen until the reagent is exhausted. Protect the reagent from the air by a toy balloon.

Exercise No. 15

PREPARATION OF OXYGEN AND FORMATION OF OXIDES (33-40)

Study the illustration of the apparatus (Fig. 7), and construct your set-up accordingly. The pyrex ignition tube should be inclined **slightly** (mouth lowest). The clamp should hold the ignition tube close to the rubber stopper. Have your set-up inspected before proceeding.

(a) Generation of Oxygen.

Mix on a piece of paper about 5 ml (8 g) of potassium chlorate with an equal amount of manganese dioxide. Insert the mixture in the pyrex tube and roll the latter until the mixture is spread over one-half the length of the tube. Replace the tube and stopper. Fill the bottles with water, cover with a glass plate, and invert in the pneumatic

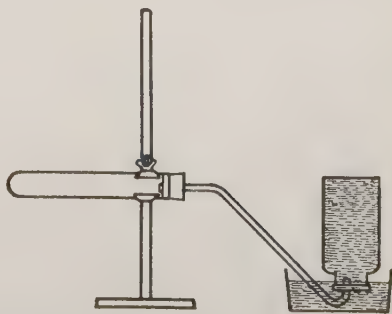


Fig. 7.—Apparatus for Preparation of Oxygen.

trough. Heat the tube gently at first, and then with the full flame of the burner. Discard the first few bubbles and fill the bottles with the gas. Before removing a bottle of gas from the trough, cover with a glass plate. When the heating is concluded, remove the end of the glass delivery tube from the water, before removing the burner.

(b) Formation of Oxides.—1. Gently heat a quantity of red phosphorus the size of a pin head in the combustion spoon, and plunge it into a bottle of oxygen. Compare the rate at which it burns in oxygen with its rate of burning in air. Write the equation. What evidence indicates that a chemical change has occurred? Name the product formed.

2. In a similar manner, insert some hot iron (steel wool) into a bottle of the gas. Describe what occurs. Write the equation. Has a chemical change occurred?

3. Repeat 2, using aluminum powder. The powder should be heated strongly before it is immersed in the oxygen. It is best to use a fresh bottle of gas. Write the equation.

4. Compare the rate at which sulphur burns in oxygen with the rate at which it burns in air. Upon what observations do you base your conclusion?

5. Clean the combustion spoon and repeat, using carbon in place of sulphur. Write the equation and name the products formed.

Exercise No. 16

PREPARATION OF HYDROGEN AND ITS USE AS A REDUCING AGENT (40-44)

Study the illustration of the apparatus (**Fig. 8**), and construct your own set-up in exact accordance therewith. Your apparatus must be inspected before proceeding. The pyrex ignition tube should be inclined slightly (mouth lowest). The copper oxide (wire form) should be near the rounded end of the tube. The bottom of the funnel tube should be about $\frac{1}{8}$ inch off the bottom of the flask.

(a) Place 10-15 g of zinc in the flask, replace the stopper, and add water, until the bottom of the funnel tube is just covered. Add sulphuric acid, 10 per cent solution, in small amounts at a time, until a vigorous evolution of gas takes place. **Before applying heat to the ignition tube, the flask must be wrapped**

in a towel, and the emerging gas tested to prove the absence of air. Otherwise, an explosion may result. The test consists of collecting test tubes of gas and igniting it. When these show the gas to be free from air, a flame may be applied to the pointed outlet tube and the hydrogen ignited. If the hydrogen burns quietly, it is safe to heat the ignition tube. The drying tube or U-tube contains calcium chloride, which removes the water vapor from the hydrogen before the latter reaches the copper oxide.

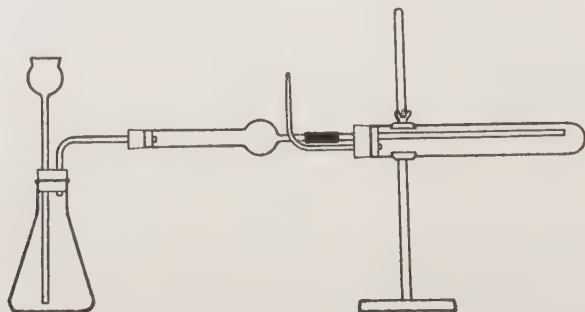


Fig. 8.—Apparatus for Preparing Hydrogen and Showing Its Property as a Reducing Agent.

Write the equation representing the reduction of copper oxide. What evidence indicates that the copper oxide has undergone a chemical change? (Study very carefully the information given under "Oxidation and Reduction," page 42 in the text.) Which is the oxidizing agent, and which the reducing agent? Which substance has been oxidized, and which reduced? What is the change in valence of the copper? Write the equation representing the replacement of hydrogen by zinc. Show how the reduction of copper oxide by hydrogen illustrates the definition of oxidation and reduction. What evidence indicates that water is produced when copper oxide is reduced by hydrogen?

Exercise No. 17

REDUCTION OF FERRIC CHLORIDE (42-44)

(a) **Tests for Tervalent Iron.**—To three separate 5 ml portions of ferric chloride solution, add three drops of the following reagents, which are used as indicators: (1) potassium sulphocyanate, (2) potassium ferrocyanide, (3) potassium ferricyanide. Tabulate the colors obtained.

(b) **Tests for Bivalent Iron.**—Repeat (a), using a solution of ferrous sulphate in place of ferric chloride. Tabulate the resultant colors, and compare them with those obtained in (a).

(c) **Reduction of Ferric Chloride.**—To 25 ml of ferric chloride, add 5 ml of concentrated HCl and a few pieces of zinc. When the solution is practically colorless, withdraw a 1 ml portion of the solution, dilute with an equal volume of water, and add a few drops of potassium sulphocyanate. Note the depth of color. Follow the progress of the reduction in this way. When the withdrawn portion ceases to give a color reaction with potassium sulphocyanate, the reduction of ferric chloride to ferrous chloride is complete.

(d) Test 5 ml portions of the reduced solution with potassium ferrocyanide and potassium ferricyanide. **What evidence convinces you that the ferric chloride has been reduced?**

(e) Write the equation representing the reduction of ferric chloride by nascent hydrogen. What was the source of the nascent hydrogen? How does the equation just written illustrate the definition of Oxidation and Reduction? What is the change in valence of the iron?

Exercise No. 18

OXIDATION OF FERROUS SULPHATE (42-44)

(a) Review the tests for bivalent and trivalent iron, as performed in (a) and (b) in Exercise No. 17.

(b) **Oxidation of Ferrous Sulphate.**—To 25 ml of ferrous sulphate solution, add 2 ml of sulphuric acid and then potassium permanganate solution in small portions at a time, with stirring, until a faint but permanent pink color remains in the solution.

(c) Test 5 ml portions of this solution with each of the reagents used in (a), Exercise No. 17. Record the resultant colors, and compare them with the colors obtained in the tests for bivalent and trivalent iron.

(d) What is the valence of the iron before and after treatment with potassium permanganate? Is potassium permanganate an oxidizing or a reducing agent? Write the formula for ferric sulphate and compare it with that for ferrous sulphate. Show how the transformation of ferrous sulphate to ferric sulphate is an example of oxidation and reduction.

Exercise No. 19

WATER AS A SOLVENT AND REAGENT (47-48)

(a) Suspend a crystal of copper sulphate by a thread in a beaker of water. Watch the process of **diffusion**. An attractive force is exerted between the water and the copper sulphate, resulting in the latter being divided into a great many small particles, each surrounded by as much water as possible.

(b) **Hydrolysis**.—To 2 ml of antimony chloride solution in a beaker, add 50 ml of water in small portions at a time. Review the definition of hydrolysis in the text and use this definition in writing the equation for the above reaction.

(c) Introduce a piece of calcium carbide (CaC_2) into 5 ml of water. Does a reaction occur? Record the evidence that substantiates your conclusion. Touch the burner flame to the mouth of the test tube. The gas is acetylene, C_2H_2 . Write the equation. Show that this equation is an illustration of **hydrolysis**.

(d) Grind some oxalic acid with an equal amount of sodium carbonate. Pour 10 ml of water upon the system. Describe the result and prove that a chemical reaction has taken place. Did the water take part in the reaction? Why did the two substances not react until the water was added?

(e) Place a small piece of sodium in a 250 ml beaker containing 25 ml of water and cover with a watch glass. Does a reaction occur? What evidence supports your conclusion? Write the equation. Does the result agree with the electrochemical series?

Exercise No. 20

STUDY OF A NATURAL WATER (48-52)

(a) Evaporate to dryness, 25 ml of water on a **thoroughly clean** watch glass over a beaker of boiling water. What is the residue?

(b) To each of two 100 ml portions of water, one of which is tap water and the other distilled water, add 5 ml of potassium permanganate solution and 2 ml of sulphuric acid. Heat to boiling. Can you observe any difference? Potassium permanganate is a strong oxidizing agent and the amount of it in solution

at any time may be estimated by noting the intensity of the color. Explain why a natural water may require more permanganate solution to produce a given intensity of color than does distilled water.

(c) To 300 ml of a turbid and colored water, add 10 ml of aluminum sulphate solution and then add ammonium hydroxide until the system **just** turns red litmus paper blue. Allow the system to stand for one hour and then filter. Compare the filtrate with the original untreated water. Explain the difference and show how your result bears on the purification of water for drinking purposes.

(d) On the side table, there has been arranged an outfit to show how water may be distilled in the laboratory. The liquid in the flask is a dilute solution of potassium permanganate. What explanation can you offer in regard to the color of the distillate?

Exercise No. 21

STUDY OF SOME ACIDS AND ACIDIC OXIDES (54-55)

Pay particular attention to the main characteristics of an acid, and to the meaning of terms.

(a) Study the acids designated¹ by the instructor with respect to (1) name, (2) formula, (3) binary or ternary, (4) basicity. Tabulate the information.

(b) Pour a few drops (small piece if the acid is solid) of each acid separately into 5 ml of water. Determine the **reaction** of each solution by conveying a drop with a glass rod to a piece of litmus paper of each color. What is the **reaction** of each solution?

(c) Taste one of the solutions, using a very small drop.

(d) Immerse a piece of zinc in 5 ml of hydrochloric acid. What is the gas? Write the equation. How does this equation illustrate one of the main characteristics of an acid? What method of chemical change does this equation represent?

(e) Put some sulphur in the combustion spoon, ignite it, and while burning, hold it inside a bottle containing 25 ml of water for several minutes. Extinguish the flame by lowering the spoon into the water. Remove the spoon, place the palm of the hand over the mouth of the bottle and shake vigorously. Determine

¹ The following acids may be used: hydrochloric, nitric, sulphuric, phosphoric, oxalic, acetic, tartaric.

the reaction of the liquid with litmus paper. Explain how the water has acquired this reaction, using two equations.

(f) Carbon dioxide is the anhydride of what acid? Using a glass tube, blow through 5 ml of calcium hydroxide solution. Write the equation.

(g) Phosphorus pentoxide (P_2O_5) is the anhydride of what acid? Add a small amount to 25 ml of water. Does it react with the water? Write the equation. Determine the reaction of the solution.

(h) The anhydride of acetic acid is $(CH_3CO)_2O$. This is a liquid anhydride. Add one drop to 10 ml of water. What is the reaction?

(i) Place 3 or 4 drops of sulphuric acid in a crucible, and heat (in hood) carefully until heavy white fumes of SO_3 appear. Keep your face away from the crucible. These white fumes are the anhydride of sulphuric acid. Write the equation representing their formation from the acid.

(j) Tabulate the properties of all the acid anhydrides which you have used, giving (1) name, (2) formula, (3) state of matter, (4) color, (5) the acid from which the anhydride may be made by removing water.

Refer to Table II in the text.

Exercise No. 22

STUDY OF SOME BASES AND BASIC OXIDES (56-59)

Pay particular attention to the two main characteristics of bases and to the meaning of terms.

(a) Study the bases designated¹ by the instructor in regard to (1) name, (2) formula, (3) state of matter, (4) acidity. Tabulate.

(b) Dissolve a very small amount of each base separately in water. Test the resulting solutions with both kinds of litmus paper. Record the reactions.

(c) Drop a very small piece of sodium upon 10 ml of water in a beaker and cover with a watch glass. Determine the reaction of the solution. Write the equation. What is the name of the base formed?

(d) **Basic Oxides.**—Add about 2 ml of hot water to a lump of calcium oxide the size of a walnut. What can you observe?

¹ The following may be used $NaOH$, KOH , NH_4OH , $Ba(OH)_2$, $Ca(OH)_2$.

Write the equation for the reaction of calcium oxide with water. Add 50 ml more of water, stir, allow to settle, and decant the supernatant liquid onto a filter paper. Test the clear filtrate with litmus paper. What base is present in solution?

(e) Add 25 ml of water to a small amount of ferric oxide in a beaker. Heat, stir, and filter. Pour the filtrate through the same filter paper several times until perfectly clear. Test the filtrate with litmus. What is the reaction? Evaporate 10 ml of the filtrate to dryness on a watch glass. Is this basic oxide soluble in water?

(f) To another small portion of ferric oxide, add 10 ml of concentrated hydrochloric acid and heat for a moment. Filter as in (e) and evaporate a portion. What evidence of a chemical reaction between the ferric oxide and the hydrochloric acid do you observe? Why is ferric oxide called a basic oxide?

(g) A soluble base may be used to prepare an insoluble base or basic oxide. To 10 ml of copper sulphate solution, add sodium hydroxide solution until the system is alkaline. Add 1 ml of the NaOH solution in excess. What is the color of the precipitate? Write the equation. Heat the test tube containing the precipitate. What is the change in color? The change in color is caused by the decomposition of the precipitated hydroxide into water and the oxide of the metal. Write the equation.

(h) What classes of compounds are formed, respectively, when metallic and non-metallic oxides react with water? In general, how may basic oxides be formed from the hydroxides of the metals?

Exercise No. 23

NEUTRALIZATION AND THE FORMATION OF SALTS (56-61)

Review the definition of an acid and a base. Study the information on pages 56 to 61 of the text, paying particular attention to the meaning of terms. Be sure you understand the meaning and application of the following terms: (1) acids and bases; (2) acidic and basic substances; (3) acid and alkaline reactions of solutions.

(a) To 10 ml of sodium hydroxide solution in a beaker, add hydrochloric acid in 1 ml portions at a time until blue litmus paper touched with a drop of the system turns red. Now add sodium hydroxide solution, **drop by drop**, until the system barely

turns red litmus blue. The hydrochloric acid and the sodium hydroxide have **neutralized** each other. Write the equation.

(b) Evaporate 5 ml of the neutral solution to dryness on a watch glass. Taste the residue. What is it?

(c) Determine experimentally whether sodium carbonate solution has an acid or alkaline reaction. Is it an acidic or basic substance? Determine this latter point by adding hydrochloric acid, drop by drop, to 10 ml of hot sodium carbonate solution, testing continuously with litmus paper until an acid reaction is obtained. Write the equation. Evaporate 10 ml of the neutral system to dryness. What is the residue?

(d) To a small piece of calcium oxide, add 25 ml of water and heat for a moment. What is the reaction of the solution? Equation. Add hydrochloric acid in small portions at a time. Does the reaction of the solution change? Equation. What salt is present in the system? Is calcium oxide an acidic or a basic substance?

(e) Treat a small piece of zinc with 5 ml of dilute sulphuric acid. Write the equation. What salt is present in solution? Is one justified in calling metallic zinc a basic substance?

(f) Treat a small amount of ferric oxide with 10 ml of hydrochloric acid and heat. Decant the supernatant liquid upon a filter paper. Has a chemical reaction taken place? What leads you to believe so? Add ammonium hydroxide to the clear filtrate until an alkaline reaction is obtained. What salt was formed when the acid reacted with the oxide? Equation. What is the composition of the system after the ammonium hydroxide was added? Equation.

(g) Summarize the methods of salt formation illustrated above. Does every case of salt formation involve the process of neutralization?

(h) Prepare some aluminum hydroxide by adding ammonium hydroxide to 15 ml of aluminum sulphate solution until an alkaline reaction is obtained. Filter. Divide the jelly-like precipitate into two portions.

1. To one portion add hydrochloric acid until a strong acid reaction is obtained. What happens? Equation.

2. To the other portion add 15 ml of sodium hydroxide solution. What happens? Equation. See text, pages 58 and 59. When does aluminum hydroxide act as an acid, and when as a base?

Exercise No. 24

PREPARATION AND PROPERTIES OF HYDROGEN PEROXIDE (76-78)

(a) **Properties of Hydrogen Peroxide.**—To 5 ml of a dilute indigo solution, add 3 ml of hydrogen peroxide and then sodium hydroxide solution drop by drop until the system is alkaline. Heat for a few minutes. Describe the result. Explain.

(b) To 10 ml of lead nitrate solution, add 10 ml of water. Pass hydrogen sulphide gas into the system for about a minute (**hood**). Allow the precipitate to settle and decant the supernatant water. Add 5 ml of hydrogen peroxide to the precipitate and heat gently. Describe the result. The black precipitate is lead sulphide, PbS , and the white substance formed after the reaction with hydrogen peroxide is lead sulphate, PbSO_4 . Write the equation to represent the reaction of the black precipitate with hydrogen peroxide. In this case, is hydrogen peroxide an oxidizing or a reducing agent?

(c) To 5 ml of hydrogen peroxide, add a small amount of manganese dioxide (finely powdered) in small portions at a time. The catalytic action of the manganese dioxide results in the decomposition of the hydrogen peroxide. Test the issuing gas with a glowing splint. What is it? Write the equation for the decomposition of hydrogen peroxide.

(d) To 5 ml of hydrogen peroxide, add 3 or 4 drops of sulphuric acid and then a solution of potassium permanganate **drop by drop**. Describe the result. Potassium permanganate is a strong oxidizing agent and is decolorized when it is reduced. Is hydrogen peroxide always an oxidizing agent?

(e) Convey a drop of hydrogen peroxide to a piece of starch-potassium iodide paper. Describe the result. Starch-potassium iodide paper is used as a general test for oxidizing agents.

(f) To 5 ml of a saturated calcium hydroxide solution, add 2 ml of hydrogen peroxide. The precipitate formed is calcium peroxide. Write the equation.

(h) To 3 ml of hydrogen peroxide, add 3 drops of sulphuric acid. Cool the system with ice. Add 4 or 5 drops of potassium dichromate and then 2 ml of ether. Shake gently. A blue color in the ether layer is a test for hydrogen peroxide.

(i) **Preparation of Hydrogen Peroxide.**—Add 5 ml (7 g) of barium peroxide to 25 ml of ice-cold water and stir. While the barium peroxide is suspended in the water, add sulphuric acid until the system is acid. The acid should be cold. Write the equation. Allow the precipitate to settle and decant the liquid onto a filter paper. Test the clear filtrate as in (d) and (e), given above.

(j) To 25 ml of cold water in a beaker, add 1 ml (1 g) of sodium peroxide in small portions at a time. Acidify with H_2SO_4 and test the system for the presence of hydrogen peroxide as in (d) and (e). Write the equation. (**Caution**—do not place sodium peroxide upon paper, as the latter may take fire.) Any unused sodium peroxide should be dissolved in water and run into the sink.

Exercise No. 25

DETERMINATION OF CHEMICAL EQUIVALENT BY REPLACEMENT (81-96)

Read Exercise No. 13 again, and note the precautions to be observed in using the analytical balance.

(a) Weigh to the nearest milligram the sample of the designated metal (Cu, Zn, Mg). Add this quantity of the metal to 50 ml of the designated solution of silver nitrate in a small beaker or evaporating dish that has been weighed to the nearest milligram. Cover the system with a watch glass and allow to stand until the next laboratory period.

(b) Before filtering, feel around the bottom of the beaker or dish with a stirring rod and assure yourself that all of the original metal has reacted with the silver nitrate solution. If it has not, add 10 ml of silver nitrate solution and allow to stand again.

Decant the liquid carefully upon a filter paper. Allow as few particles as possible of the precipitated metal to leave the beaker, and do not allow the liquid being filtered to rise over the rim of the paper.

Wash twice by **decantation**. This is performed as follows: add 25 ml of distilled water to the precipitate, stir, allow the precipitate to settle, and decant the liquid upon the same filter paper. When you have washed twice by decantation, collect 5 ml of the filtrate as it comes from the funnel and add 1 ml of hydrochloric acid. If no cloudiness results, the precipitate has been washed sufficiently.

(c) Place the beaker and its contents, and the funnel with the filter paper in a drying oven¹ for about a half hour. **Dry thoroughly.** Burn the paper upon an inverted crucible cover and add the residue to the beaker. Weigh the beaker to the nearest milligram.

(d) Calculate the weight of silver by difference. Knowing the weight of the designated metal and the weight of the silver replaced by it, calculate the amount of silver which would be replaced by one symbol weight of the designated metal. What does this weight of silver represent?

(e) Write the equation for the replacement of silver by the designated metal. What is the definition of the "equivalent of an element?" Calculate the amount of silver equivalent to the amount of the designated metal which you used and compare the calculated value with your experimental result by finding the per cent of error. This is a measure of your skill and carefulness.

Exercise No. 26

DETERMINATION OF CHEMICAL EQUIVALENT BY THE EUDIOMETER METHOD (81-96)

Study carefully the meaning of "**chemically equivalent**" and "**equivalent of an element**" on page 94 in the text. Review Exercise No. 13, noting the precautions necessary in using the analytical balance.

(a) Various types of apparatus may be employed in this experiment: (1) Klug's design, as illustrated in **Fig. 9**, consists of a small Erlenmeyer flask to which a side tube is attached at the bottom. To this a thistle tube is connected by a short length of rubber tubing fitted with a pinch-cock. The flask is placed in a 250 ml beaker and an inverted eudiometer tube is placed over the mouth of the flask. (2) Exner's modification (**Fig. 10**) consists of a 200 ml Erlenmeyer flask fitted with a two-hole stopper bearing a straight tube and a tube bent into a right angle. The straight tube extends to an eighth of an inch from the bottom of the flask and protrudes above the stopper about two inches. To it is attached a thistle tube by means of a short length of rubber tubing equipped with a pinch-cock. The right angle tube is just flush with the lower surface of the stopper. To it is attached a short

¹ The drying may be carried on high above a burner flame.

length of rubber tubing (8 or 10 inches) with a piece of bent tubing in the end. The end of this bent tube is inserted in the mouth of a eudiometer inverted in a beaker of water. (3) Eglof's design, represented by **Fig. 11**, consists of a 400 ml beaker in which is placed a stemless funnel. Through a deep groove which has been formed on the rim of the funnel passes the lower end of a separa-

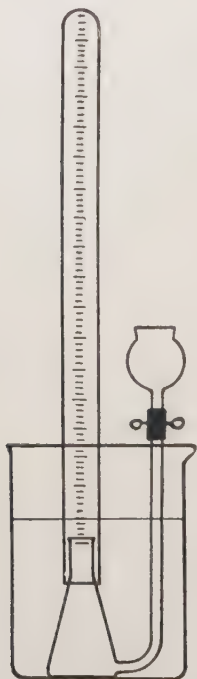


Fig. 9.—Apparatus for Determining Chemical Equivalent by the Eudiometer Method. Klug's Design.

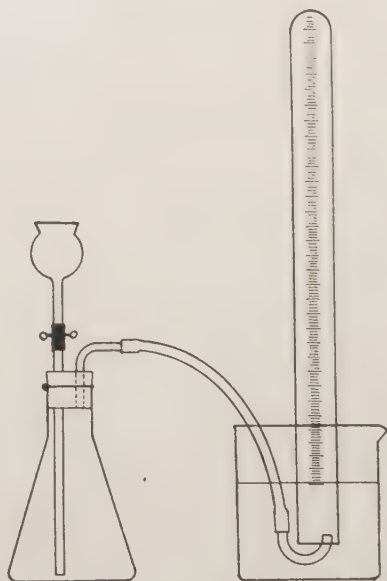


Fig. 10.—Apparatus for Determining Chemical Equivalent by the Eudiometer Method. Exner's Modification.

tory funnel bent at right angles (a thistle tube and a pinch cock may be employed). An inverted eudiometer tube rests on the apex of the funnel.

(b) Weigh¹ a sample of 20-mesh zinc or of magnesium ribbon or sheet aluminum sufficient to give enough hydrogen to displace about three fourths of the water in the endiometer tube. Place the sample in the apparatus. (The magnesium ribbon and aluminum must be wrapped in a piece of copper gauze to keep

¹ In order to insure accurate results, the instructor may provide weighed samples.

them from floating. If the third type of apparatus is used, the zinc must be placed in a shallow flat-bottomed dish made by cutting off the bottom of a litmus paper vial.) Fill the apparatus with water and free it of air by running water through it. Then fill the eudiometer with water and invert it over the opening from which the gas will issue. Be sure that there is no air in the eudiometer.

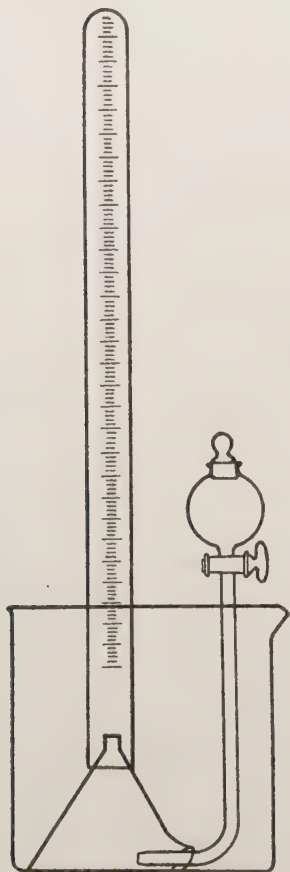


Fig. 11.—Apparatus for Determining Chemical Equivalent by the Eudiometer Method. Egloff's Design.

(c) Pour concentrated hydrochloric acid into the thistle tube, and allow it to run into the apparatus, judging the amount of acid to add by the vigor of the evolution of the hydrogen. When the evolution of gas has ceased, see that all of the hydrogen has been transferred to the eudiometer tube. This is accomplished by running water into the apparatus through the thistle tube. Cover the end of the eudiometer tube with the index finger under the surface of the solution, and transfer the tube to a tall cylinder of water, keeping the end tightly covered until the end of the tube is under the surface of the water in the cylinder. Support the eudiometer with a clamp, and adjust the height of the tube in the cylinder until the water levels inside and outside the tube are the same. Read the volume of gas in the tube. Read the temperature of the room and the barometric pressure. All gases measured over water must have their pressure corrected for the pressure of the water

vapor. Ask instructor about this. Each recorded reading must be clearly labeled so that, later, there will be no doubt as to what the readings represent. Tabulate the data.

(d) Pages 86, 87, 88 in the text should be carefully studied, together with the problems therein illustrated. From the data obtained in the above experiment, calculate the following:

(1) The grams of hydrogen gas which were liberated by the weight of sample taken. (2) The weight of the sample necessary to give you this weight of hydrogen. This should agree with the actual weight of the sample taken. Calculate per cent of error. (3) The grams of sample which are equivalent to eight grams of oxygen. (4) The number of grams of hydrogen which would have been liberated if a symbol weight of the sample had been used in place of the weight of sample actually employed. All calculations must be recorded in the notebook.

Exercise No. 27

PREPARATION AND PROPERTIES OF CHLORINE (115-118)

(a) Study the figure of the apparatus (**Fig. 12**) and construct your own in accordance therewith. Introduce about 15 ml (15 20 g) of lump manganese dioxide into the flask and replace the stopper.

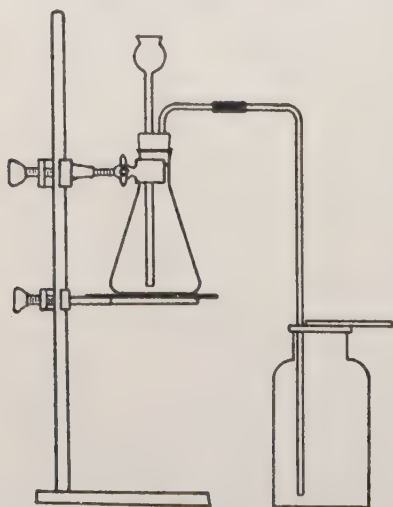


Fig. 12.—Apparatus for the Preparation of Chlorine.

Add 50 ml of concentrated hydrochloric acid. Shake the flask with a gentle rotary motion and heat (**hood**). Collect four bottles of the gas by displacement of air. When the desired number of bottles of gas have been collected, place the end of the outlet tube in a solution of sodium hydroxide to prevent the escape of the gas into the air.

(b) Write the equation representing the preparation of chlorine gas. What are some of the physical properties of chlorine gas?

(c) Suspend a piece of moist blue litmus paper in a bottle of the gas for 2 minutes. Will ammonium hydroxide restore the color? Repeat, using a piece of moist calico cloth. Describe what happens.

(d) Using the combustion spoon, heat gently some red phosphorus (a quantity about the size of a pinhead) and insert it in

the bottle of gas used in (c). Write equation, and describe what happens.

(e) Take a pinch of powdered antimony between the thumb and finger, and sprinkle into a bottle of the gas. Describe what happens, and write the equation.

(f) Moisten a piece of filter paper with turpentine, and plunge it into a fresh bottle of the gas. Describe what happens.

(g) Introduce some red hot copper turnings into a bottle of the gas. Write equation, and describe what happens.

(h) Pour 2 ml of ferrous sulphate solution into a bottle of the gas. Add 10 ml of water, and shake for a few minutes. Determine whether the iron is now in the bivalent or trivalent state by testing with potassium ferrocyanide solution.

Exercise No. 28

PREPARATION AND PROPERTIES OF HYDROCHLORIC ACID (119-123)

(a) Study the figure of the apparatus (**Fig. 12**), and construct your own in accordance therewith. Introduce about 25 ml (20-30 g) of sodium chloride into the flask and replace the stopper. Pour 15 ml of concentrated sulphuric acid down the funnel tube, and shake the flask with a gentle rotary motion until the salt is thoroughly wet with the acid, and the end of the funnel tube is covered. Heat gently (hood), and collect four bottles (must be dry) of the gas. When the last bottle has been filled, place a beaker of water so that the end of the delivery tube dips therein and the escape of the gas into the air is prevented. Give equation.

(b) Uncover one of the bottles and allow some of the gas to escape into the air. What do you observe? What are some of the physical properties of hydrochloric acid gas?

(c) Invert one of the bottles of gas with its mouth just below the surface of the water in a large beaker. What do you observe? Remove the bottle and test the reaction of the solution in the beaker with litmus paper. Explain why the water has acquired this reaction.

(d) By means of a glass rod, introduce a piece of moist blue litmus paper into a bottle of the gas. What happens? Moisten a piece of filter paper with concentrated ammonium hydroxide and immerse in a bottle of the gas. What happens? Write the equation,

(e) **Test for a Chloride.**—To 5 ml of a solution of hydrochloric acid, add a few drops of silver nitrate solution. This precipitate is characteristic and is proof of the presence of chlorides in a system. Solutions which have an alkaline reaction should be acidified with nitric acid and boiled, before testing for the presence of chlorides.

(f) Treat a small piece of zinc with 5 ml of hydrochloric acid. Write the equation. Treat a small amount of sodium carbonate with 5 ml of hydrochloric acid. Write the equation. **What evidence have you observed throughout this entire experiment which indicates that hydrogen chloride is an acid?**

Exercise No. 29

THE CHLORIDES OF THE ALKALI GROUP (123-130)

(a) Write the formulas for the chlorides of the alkali group. Examine a few crystals of each. Dissolve a little of each separately in 5 ml of water. Determine the reaction of each resulting solution with litmus paper.

(b) Heat strongly a small portion of each upon an inverted porcelain crucible top. Describe any differences in behavior. Which salts decrepitate?

(c) Fill the rounded portion of a test tube with ammonium chloride and heat strongly. Does the salt melt? Describe the behavior of the system. Write the equation showing the decomposition of ammonium chloride by heat and show how this equation illustrates the process of dissociation.

(d) Thoroughly clean a platinum wire by repeated dipping into 1 ml of concentrated hydrochloric acid in a watch glass, and then holding the wire in the flame. Change the acid frequently. The wire is clean when it will not color the flame after dipping in fresh acid. (Do not dip the wire into the reagent bottle.)

Bend a small loop on the end of the wire, moisten with water and pick up a small crystal of each salt separately. Hold in the outer edge of the flame near the burner top. Describe the characteristic colors produced when sodium and potassium compounds are volatilized in the flame. If both sodium and potassium occur in the same system, it is possible to recognize potassium by viewing the flame through a blue glass plate. Try the flame reaction of a mixture of the two salts. (See table in Appendix.)

Exercise No. 30**THE CHLORIDES OF THE ALKALINE EARTH GROUP
(130-131)**

(a) Examine the hydrated chlorides of the alkaline earth group. Write the formulas. Determine whether each is soluble in water. Try the reaction of the water solutions with litmus paper.

(b) Make a saturated solution of magnesium chloride by adding the salt in small portions at a time to 5 ml of water until no more will dissolve. Heat in a small beaker covered with a watch glass on the under side of which is pasted a piece of moistened red litmus paper. Observe the litmus paper. Explain. What is the reaction? Explain with the aid of an equation. Is this a case of hydrolysis?

(c) Fill the rounded portion of a test tube with magnesium chloride and heat. How do you explain the presence of so much moisture? What is meant by "water of crystallization"? A hydrate? An anhydrous salt?

(d) Allow a small piece of calcium chloride to stand on a watch glass during the laboratory period. What is deliquescence?

(e) Determine the flame reactions of the members of this group as was done in (d) in **Exercise 29**. Record. (See table in Appendix.)

Exercise No. 31**THE CHLORIDES OF THE ALUMINUM GROUP (131-132)**

(a) Examine the hydrated chlorides of this group. Write the formulas. What are the characteristic colors? Determine whether these chlorides are soluble in water by adding a small amount of each to 5 ml of water. Test the reaction of each solution with litmus paper. Record the reactions.

(b) Saturate 5 ml of water with aluminum chloride, heat, and hold a piece of blue litmus paper in the vapors coming from the solution. What is the reaction? How is this experiment an illustration of hydrolysis? Explain with the aid of an equation.

(c) Add ammonium hydroxide to 10 ml of ferric chloride solution until an alkaline reaction is obtained. Write the equation.

What is the name of the precipitate? Now, add hydrochloric acid until the system has an acid reaction, then add 2 ml in excess, and heat. What happens? Write the equation. Now, with the aid of the two equations just written, study the equation representing the hydrolysis of ferric chloride. **Why does not a solution of ferric chloride hydrolyze completely and cause the appearance of the dark red precipitate?** See "Equilibrium" in the text, page 108.

Exercise No. 32

THE CHLORIDES OF THE ZINC GROUP (132)

(a) Examine the hydrated chlorides of this group, and prepare a table giving the name, formula, and the color of each salt.

(b) Dissolve a little of each salt in 5 ml of water, and test the reaction of each solution. Write equations illustrating the hydrolysis of each salt. Explain the reactions of the water solutions with litmus paper.

(c) Place 2 ml of cobalt chloride solution in the evaporating dish, and evaporate carefully to dryness. Continue heating for a few minutes. Allow to cool, and add a few ml of water. Describe, and explain what occurs. Using a solution of cobalt chloride, write your name on a piece of paper. Dry this and heat carefully. Explain.

(d) Repeat (c), using a solution of nickel chloride.

(e) What is the valence of the elements in this group? Write the formulas for the oxides. Write equations illustrating the preparation of the chlorides from the carbonates, using hydrochloric acid.

Exercise No. 33

THE CHLORIDES OF THE ARSENIC GROUP (132-133)

(a) Examine the chlorides of the metals of this group. Write their names, formulas, states of matter, and colors. Write equations for preparing them directly from the elements.

(b) Put a small amount of antimony trichloride in a test tube. Add water, and shake. Does it dissolve? Is there any evidence of a chemical reaction? Repeat, using arsenic and bismuth chlorides.

(c) To a small amount of antimony trichloride, add 5 ml of concentrated hydrochloric acid and shake until dissolved. To 100 ml of water in a beaker, add a few drops of the solution in the test tube. Describe the result.

(d) What is the definition of hydrolysis? Show how the reaction of antimony trichloride and water illustrates this definition.

Exercise No. 34

THE CHLORIDES OF THE COPPER GROUP (132-134)

(a) Examine the chlorides of the copper group, and write their names and formulas.

(b) To 15 ml of normal lead nitrate solution in a beaker, add concentrated hydrochloric acid until no more precipitate forms. Filter, and throw away the filtrate. Wash the precipitate with 10 ml of cold water acidified with HCl. Examine the precipitate on the filter paper. What is its name and formula? Write the equation illustrating its formation.

Pour 5 ml of **boiling** water on the precipitate on the filter paper, and catch the filtrate in a small beaker. Cool the beaker and examine its contents. Record your observations. Can you observe any difference in physical properties between the substance on the filter paper and that in the beaker? What are some characteristic properties of lead chloride?

(c) Dissolve a little hydrated cupric chloride in 5 ml of water. Test the reaction of the solution with litmus paper. Pour the solution into an evaporating dish and evaporate to dryness. Describe the color changes. Cool and add water. What are the resulting color changes? How do you explain them?

Add ammonium hydroxide drop by drop, with stirring, until the system become homogeneous. Describe the behavior of the system, noting any changes in color and the formation of precipitates.

(d) To 5 ml of water, add a little stannous chloride. Does it dissolve? If not, add 2 ml of concentrated hydrochloric acid and heat gently. If it does not dissolve with this treatment, add a small piece of metallic tin and heat again. Prepare a solution of stannic chloride, by adding a small amount of the salt to 5 ml of water.

Add 1 ml of sodium sulphide solution to 5 ml of each of the two solutions just prepared. Note the colors and form of the precipitates and write equations showing their preparation.

(e) Heat a few crystals of mercuric chloride in a dry test tube. Do not breathe any vapor issuing from the tube. Describe the behavior of the system. Allow the tube to cool, and add 5 ml of water. Divide the solution into two portions. Add 2 ml of stannous chloride to one portion, and the same amount of stannic chloride to the other. Describe what happens. Write equations. Show how the reaction of stannous chloride with mercuric chloride is an illustration of oxidation and reduction. Which is the oxidizing agent, and which the reducing agent?

Exercise No. 35

THE CHLORIDES OF THE SILVER GROUP (134)

(a) Place 3 ml of cupric chloride and an equal quantity of metallic copper in a small beaker. Add 20 ml of concentrated hydrochloric acid, and boil for 20 minutes, replacing the acid as it evaporates. Filter, and pour the filtrate into a large beaker of cold water. Allow to settle, decant, then transfer the precipitate to a filter paper, and wash. What is the color of the precipitate? Write the equation showing its preparation.

Transfer the precipitate to a small beaker, add 5 ml of ammonium hydroxide and heat. Does cuprous chloride react with ammonium hydroxide? What indicates this?

(b) To 5 ml of mercurous nitrate solution in a beaker, add hydrochloric acid drop by drop until no more precipitate forms. This may be determined by allowing the precipitate already formed to settle, and then adding a drop of the precipitating reagent to the surface of the solution. If no further precipitation occurs, sufficient hydrochloric acid has been added. Heat the solution and precipitate for a moment, filter and wash, and discard the filtrate. Write the equation for the reaction between mercurous nitrate and hydrochloric acid. What are the two products formed? Which is the precipitate? What happens to the other product?

Pour some ammonium hydroxide on the precipitate on the filter paper. What happens? Does the precipitate react with ammonium hydroxide?

(c) Repeat the first paragraph in (b), using silver nitrate in place of mercurous nitrate. Pour 5 ml of ammonium hydroxide on the precipitate on the filter paper. Catch the filtrate in a beaker and add nitric acid until an acid reaction is indicated with litmus paper. Does silver chloride react with ammonium hydroxide? Write the equation for its preparation from silver nitrate. Transfer the precipitate thrown down by nitric acid to a filter paper and expose to the sunlight. What happens?

(d) Tabulate the properties of the three chlorides of this group in regard to name, formula, solubility in water, and behavior in ammonium hydroxide.

Exercise No. 36

BROMINE AND IODINE (135-139)

(a) Grind 2 ml (2 g) of sodium bromide with an equal amount of manganese dioxide in the mortar. Transfer to a test tube, and add 5 ml of dilute sulphuric acid. Heat cautiously **in the hood**. The red fumes are bromine vapor. Oily drops of bromine may collect upon the side of the test tube. Be careful about breathing any of the vapor or allowing any of the liquid bromine to touch the skin. It is best to wash your hands after handling the tube. The reaction takes place in two steps. The sulphuric acid produces hydrobromic acid which is oxidized to bromine by the manganese dioxide. Write the system of equations. What are the physical properties of bromine?

(b) Examine a bottle of iodine crystals. What are the physical properties? Repeat (a) using sodium iodide in place of sodium bromide. Heat a small crystal of iodine in a test tube. Describe the result. What is sublimation? Write two systems of equations showing the preparation of iodine from sodium or potassium iodide.

(c) Grind 1 ml (1 g) of starch with 10 ml of cold water. Pour the resulting paste into 100 ml of boiling water. Stir and heat for a few minutes. This is called "starch solution." To 10 ml of water, add 2 ml of the starch solution, and two drops of a solution of iodine. The resulting color is an important test for iodine.

Add a few drops of bromine water to another 5 ml portion of the starch solution. Compare the color with that produced by iodine.

(d) To 5 ml of potassium iodide solution in a test tube, add 1 ml of chlorine water. Add 2 ml of carbon tetrachloride to the test tube and shake gently. Describe the appearance of the system. Explain the color of the carbon tetrachloride layer.

(e) Repeat (d), using a solution of bromine in water in place of the chlorine water. Write the equation for the reaction of bromine upon potassium iodide. Study the results of (d) and (e) in the light of the order of bromine and iodine in the electrochemical series.

(f) Iodides are frequently used as reducing agents. To 5 ml of starch solution, add 1 ml of potassium iodide solution, and then a few drops of ferric chloride solution. Explain the resulting color and write the equation illustrating the reduction of ferric chloride by potassium iodide. Show why the liberation of iodine from potassium iodide constitutes oxidation of the latter salt.

(g) By the same method employed in (f), determine whether a solution of sodium nitrite which has been acidified with hydrochloric acid will oxidize a solution of potassium iodide.

Exercise No. 37

PREPARATION AND PROPERTIES OF HYDROGEN SULPHIDE (145-146)

(a) Examine carefully the hydrogen sulphide generator as arranged by the instructor, and construct your own in accordance therewith. Introduce into the generator about 25 g of ferrous sulphide and then add 40 to 50 ml of the sulphuric acid provided for the purpose. Pass hydrogen sulphide into the following:

(b) **Water.**—Is the gas soluble in water? Test the solution with both kinds of litmus paper. What is the reaction to litmus paper? Observe the color and odor of the gas. Record.

(c) **Sodium Hydroxide Solution.**—Is the gas absorbed by a solution of sodium hydroxide? What two compounds are formed when sodium hydroxide reacts with hydrogen sulphide? Write the equation and compare it with the equation representing the neutralization of sodium hydroxide by hydrochloric acid. Name hydrogen sulphide as a binary acid. (See sodium sulphide reagent among Reagents in Appendix.)

(d) **Ammonium Hydroxide.**—What is the color of the solution obtained when hydrogen sulphide is passed into 25 ml of a solution

of ammonium hydroxide? Write the equations. Add 2 ml of powdered sulphur to the solution and continue to pass in hydrogen sulphide. Equation? Does the sulphur dissolve? What evidence indicates this? What are polysulphides? Filter the solution which has been treated with sulphur, and to 5 ml of the filtrate, add hydrochloric acid until an acid reaction is obtained with litmus paper. What is the precipitate? Equation? Save the remainder of the filtrate for use in (e).

(e) **Solutions of Lead Nitrate, Arsenic Chloride, Antimony Trichloride, Cadmium Chloride, Manganese Chloride, and Zinc Chloride.**—Use 2 ml of the solutions provided and dilute to 10 ml with water.

Write the equations for the reactions of hydrogen sulphide with the above salts. Give the colors and names of the precipitates formed. Is antimony sulphide soluble in yellow ammonium sulphide solution? In sodium sulphide reagent? Test by using a small quantity of the precipitate.

(f) **Ferric Chloride.**—To 5 ml of water in a test tube, add a few drops of hydrochloric acid, and then add 2 ml of ferric chloride solution. Pass hydrogen sulphide into the solution for 2 minutes. Do you observe any change? Filter the solution through the same filter paper repeatedly until clear. Determine whether the iron is now in the bi- or ter-valent state. See Exercises Nos. 17 and 18. Is hydrogen sulphide a reducing or an oxidizing agent? Express all reactions by equations.

(g) Using the blow-pipe or a pointed glass tip, burn the hydrogen sulphide in the air. Observe the odor of the combustion products. What are they? Write the equation which represents the complete combustion of hydrogen sulphide.

Exercise No. 38

SOLUBILITY OF SULPHIDES IN ACID AND ALKALINE SOLUTIONS (145-149)

(a) To 10 ml of CuSO_4 solution in an Erlenmeyer flask, add 10 ml of H_2O and then add 2 ml of concentrated HCl . Fit the flask with a one-hole stopper carrying a delivery tube reaching to within $\frac{1}{4}$ inch of the bottom. Connect this delivery tube to the H_2S generator. Pass H_2S directly into the solution (with shaking) until precipitation is complete. This is determined by allowing

the precipitate to settle, and then decanting 2 or 3 ml of the supernatant liquid upon a filter paper, catching the filtrate in a test tube, and treating it with H_2S . If no precipitate forms, the precipitation in the original flask is complete.

Filter the solution containing the copper sulphide, paying particular attention to the details of filtration. (See page 6.) Wash the copper sulphide on the filter paper until the wash water coming through the paper is neutral to litmus.

Using the horn spoon or spatula, divide the precipitate into four portions, and transfer each to a small beaker or evaporating dish. Add 10 ml of concentrated HCl to one portion, 10 ml of concentrated nitric acid to the second, 10 ml of ammonium hydroxide to the third, and 10 ml of sodium sulphide reagent (Appendix) to the fourth. Heat each portion for a few minutes, and determine the behavior of copper sulphide with each of these reagents. Equations. Summarize the properties of copper sulphide.

(b) Repeat (a) in all details, using 5 ml of the antimony trichloride solution in place of copper sulphate. If, at the beginning, a white precipitate forms when water is added, add concentrated HCl , with stirring, until the white precipitate disappears. Then pass in H_2S and continue. Filter that portion in which antimony sulphide was treated with the sodium sulphide reagent and then treat the filtrate with HCl until an acid reaction is obtained with litmus paper. The precipitate is antimony sulphide. Summarize the properties of antimony sulphide. Equations.

(c) To 10 ml of ZnSO_4 in a beaker, add 10 ml of water and then 2 ml of hydrochloric acid. Pass in H_2S . Is a precipitate formed? Now add ammonium hydroxide until a faint alkaline reaction is obtained with litmus paper. Pass in H_2S again. Is a precipitate formed? Equations. How may copper and zinc be separated from each other when they occur in the same solution?

(d) Study the effect of passing H_2S into a solution of calcium chloride when the system is acid, when it is neutral, and when alkaline. Is a precipitate formed?

(e) **Devise a scheme of analysis in which copper, zinc, and calcium may be separated from each other by taking advantage of the solubilities of their sulphides in acid or alkaline solutions.**—Study the solubilities of the sulphides with respect to the position of the metal in the electrochemical series.

Exercise No. 39

PREPARATION AND PROPERTIES OF AMMONIA (152-153)

(a) Study the sample apparatus arranged by the instructor, and construct your own in accordance therewith. The dry bottles, glass plates and other required apparatus should be in a convenient place before beginning.

(b) Mix 20 ml of powdered slaked lime with half this quantity of ammonium chloride in a mortar or on a piece of paper. Rub gently, and hold a piece of moistened red litmus paper about a half inch above the mixture. Observe the odor of the gas. Write the equation for the reaction of slaked lime with ammonium chloride.

(c) Transfer the mixture to the apparatus, heat, and collect four bottles of the gas. See page 8 on collection of gases. Why are the bottles held in an inverted position? What are some of the physical properties of ammonia gas?

(d) Insert one of the bottles of gas in a large beaker containing water more than sufficient to fill the bottle. Allow to stand. What happens? Explain. Test the water in the beaker with red litmus paper. Explain, using an equation.

(e) Place 1 ml of concentrated HCl in a bottle and place upon this an inverted bottle of ammonia gas. Now invert the entire system. Describe the result and write the equation. What is the salt produced?

(f) To 10 ml of concentrated ammonium hydroxide, add HCl until a neutral reaction is obtained. Evaporate to dryness, and divide the dry salt into two portions. Transfer a portion to a 50 ml beaker, add 5 ml of sodium hydroxide solution, and cover with a cover glass on the under surface of which is pasted a piece of moistened red litmus paper. Heat and observe the litmus. Explain. Remove cover glass and observe odor. Write the equation for the reaction of sodium hydroxide with an ammonium salt.

(g) Heat a small amount of ammonium sulphate in a test tube. Determine the reaction of the issuing gas, using a piece of litmus paper supported on a glass rod. Pour the liquid remaining in the tube into 10 ml of water in a small beaker and determine its reaction with litmus paper. What do these two tests with litmus paper show about the effect of heat on ammonium sulphate?

Exercise No. 40

SYSTEMS, COLLOIDAL SOLUTIONS, AND DIALYSIS (163-171)

(a) *Systems*.—From your previous experiments, select examples of different types of systems, (1) heterogeneous, (2) homogeneous. Now name three systems which you would designate solutions. Why are these so designated? Distinguish between the solvent and solute in each case. Name examples of the following types of two phase systems you have used (1) gas-liquid, (2) liquid-liquid, (3) liquid-solid.

(b) *Colloidal Solutions*.—1. To about 10 ml of warm water add a small piece (2 sq. inches) of gelatin, and warm until homogeneous. Allow to stand in ice until cool, and examine. What happens? Define sol. Define gel. Warm this again. What is the effect of change of temperature on this system?

2. To 5 ml of a concentrated solution of water glass (sodium silicate) add a few drops of HCl. Shake thoroughly. Warm a portion of this. How does this differ from the gelatin-water system?

3. Add half a test tube of water to about a gram of starch in a mortar, and grind intimately. Transfer equal portions to three test tubes. Heat about 100 ml of water to boiling, and add the contents of one tube to the boiling water. Stir, allow to cool, and filter. Filter the contents of one of the unheated tubes through another filter. Test the filtrates in each case with iodine solution for the presence of starch. Results? In which of these two systems can the constituents be separated by filtration? Explain. Heat the contents of the third tube. Results?

(c) *Dialysis*.—To 100 ml boiling water in a beaker, add 1 ml of a 33 per cent solution of FeCl_3 . A sol of beautiful reddish brown color is obtained. Cool, and place most of this in a dialyzer,¹ which consists of a piece of parchment membrane stretched tightly over the end of a tube or vessel. Submerge this in distilled water until the surfaces of the liquid inside and outside of the dialyzer are at the same elevation. Allow to stand until next period, with frequent renewals of the water on the outside. Test the water on the outside for ferric iron and for chlorine. What is inside the dialyzer? Taste it. What kind of a reaction is involved here. Represent it by an equation.

¹ See Fig. 25 in text.

Exercise No. 41**SOLUBILITY OF GASES, LIQUIDS, AND SOLIDS IN WATER
(173-180)**

(a) Recall your experimental work on the properties of HCl, NH_3 , H_2S , H_2 and O_2 . What were your methods for the collection of these gases? Why? Recall the tests you made illustrating the solubilities of these gases. What is their relative solubility?

(b) Place about 5 ml of water into each of 5 test tubes. Into one add one ml alcohol and shake. Is the system clear? Successively add one ml portions until you have added 6 ml. What is the final result? What would you state concerning the mutual solubility of water and alcohol?

(c) To the contents of the second test tube, add acetone in a similar manner. Explain the character of the system after each addition.

(d) To the contents of the third test tube, add **one** drop of aniline and shake. Result? Continue the addition of aniline drop by drop, and explain the character of system after each addition. (Ether may be employed in place of aniline.)

(e) To the contents of the fourth, add benzene, and to the fifth, add carbon tetrachloride. Classify the above pairs of liquids as (1) consolute liquids, (2) partially miscible liquids, and (3) nonmiscible liquids.

(f) To 15 ml of a normal solution of $\text{Pb}(\text{NO}_3)_2$, add 5 ml of water and 2 ml of HCl. Filter, and wash several times with cold water acidified with HCl. Treat the precipitate on the filter paper with 10 ml hot water and collect the solution in a test tube. Cool under the tap. Explain.

Exercise No. 42**ELEVATION OF THE BOILING POINT (191-193)**

Suspend a thermometer so that it is dipping into about 100 ml of water in a beaker or Erlenmeyer flask. Determine the boiling point under atmospheric conditions (pressure). This is the equilibrium temperature between the liquid and vapor at this pressure. Add to the water 10 ml (grams) of NaCl and determine the boiling point. Add 10 ml (grams) more, and redeter-

mine the equilibrium temperature. Repeat adding 10 ml (grams) of NaCl a third time. What relation exists between the concentration of the solution and the boiling point of the solution? Show by a temperature-concentration diagram.

Exercise No. 43

DEPRESSION OF THE FREEZING POINT (193-195)

Into a small beaker (100 ml) which is surrounded by cotton packed into a large beaker, introduce about 20 ml distilled water. Add snow, or finely pulverized ice, and stir. Determine the equilibrium temperature of the ice and water by a thermometer suspended in the center of the water and ice. Now into this introduce 1 ml of HCl solution provided for this purpose. Stir, and establish the equilibrium temperature. Repeat twice, using 1 ml each time. What relation have these freezing points to the concentrations? Show by a concentration-temperature diagram.

Exercise No. 44

OSMOSIS AND OSMOTIC PRESSURE (195-197)

(a) Cut the top off a carrot and make a hole in it about two inches deep and half an inch in diameter. Round the hole by means of a cork borer so that a rubber stopper will fit. Fill the cavity with a 25 per cent cane sugar solution and insert a stopper carrying a tube of small bore about two feet long. Scrape the skin from the carrot and submerge it in a beaker of water. Support the carrot by means of a clamp. Mark the level of the liquid within the tube as soon as it becomes visible and measure the rate at which it rises. Make measurements every twenty minutes for about two hours. An extension may be put on and the apparatus permitted to stand until the next period.

Optional.—Wrap a piece of parchment paper over the funnel of a thistle tube and wind thread about it tightly, to be sure that it will not leak. Or use a dialyser tube as in Exercise No. 40. Fill with a 25 per cent sugar solution, and insert a stopper carrying a capillary tube. Be sure there are no air bubbles in the apparatus, and have the liquid stand about two inches above the funnel portion. Immerse in a beaker of water, having the liquid inside

and outside at the same level. Mark the position of the column of the solution in the tube. Allow to stand for about two hours measuring the height of the column of water every half hour. Then leave it until next period and read again.

(b) Into a 50 ml beaker, pour about 20 ml of a solution of water glass (sp. gr. 1.1). Drop into this one small fragment of each of the following salts, MgCl_2 , FeCl_3 , $\text{Cu}(\text{NO}_3)_2$, MnCl_2 , NiCl_2 , $\text{Pb}(\text{NO}_3)_2$, CoCl_2 , and $\text{KCr}(\text{SO}_4)_2$. Allow to stand for some time. Observe it closely from time to time for a period of two hours. State your observations and explain fully.

Exercise No. 45

RELATIVE CONDUCTANCE OF SOLUTIONS (199-203)

(a) By means of the electrolytic cell provided (**Fig. 13**), classify the following systems as conductors and non-conductors: H_2O , $\text{C}_2\text{H}_5\text{OH}$, and water solutions of the following: KOH , $\text{C}_3\text{H}_5(\text{OH})_3$, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, H_2SO_4 , KCl . This may be done by bringing the systems between the electrodes of the cell. By means of the light bulb, it is readily ascertained whether a current passes and therefore whether the system is a conductor of electricity.

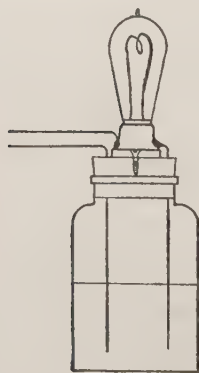


Fig. 13.—Apparatus to Show the Electrical Conductance of Solutions.

(b) Employing the following acid solutions which are approximately of the same normality ascertain the relative conductance by observing the deflection of the galvanometer needle: H_2SO_4 , HNO_3 , HCl , Acetic acid (CH_3COOH). Tabulate the acids in the order of their conductance.

(c) Similarly, determine the relative conductance of the normal solutions of the following bases and tabulate the results, KOH , NH_4OH , NaOH , $\text{Ca}(\text{OH})_2$.

(d) In a like manner, determine the relative conductance of normal solution of five of the following salts, and tabulate them in the decreasing order of their conductance: NaCl , NH_4Cl , BaCl_2 , CaCl_2 , MnCl_2 , HgCl_2 , Na_2SO_4 , MgSO_4 , MnSO_4 , ZnSO_4 , CuSO_4 .

(e) Classify the substances used in (b), (c), and (d) in two general divisions, namely, (1) strong electrolytes, and (2) weak electrolytes.

Exercise No. 46

CONCENTRATION OF SOLUTIONS—ACIDIMETRY AND ALKALIMETRY (186-189)

Acidimetry and alkalimetry comprise the processes by which the quantitative relations between acids and alkalis may be established. This is usually accomplished by establishing the volumetric relation between the solutions by the process termed titration. This consists in running into a definite quantity of an unknown solution a certain quantity of a known solution which will just neutralize the unknown solution and from this data the concentration of the unknown solution can be calculated.

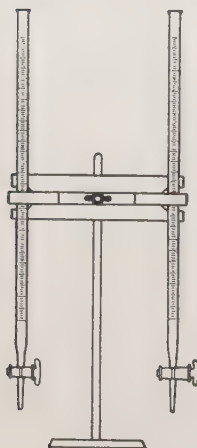


Fig. 14.—Burettes and Burette Holder.

(a) Thoroughly clean two burettes (Fig. 14) so that the water will run from these, and not remain in drops on the inside, and rinse each with about 5 ml of the solution to be used in it. Fill one burette with the known acid solution, and the other with the unknown alkali solution. Bring the under surface of the meniscus (surface of the liquid) just to the zero mark. Now run into an Erlenmeyer flask approximately 20 ml of the unknown alkali. Add about 5 drops of methyl orange indicator, and dilute with about 25 ml H_2O . From the acid burette (see that the meniscus is at zero) add rapidly about 15 ml of the known acid. Now add the acid slowly a little at a time and, finally, drop by drop, with frequent shaking, until a color change is noticed. Then add a drop or two of the alkali to bring the solution just back to alkaline or to the first appearance of the yellow. Then add acid, a drop at a time, until a golden tint appears. Wash the inside surface of the flask with distilled water, and, if the color changes, bring it back to the golden brown. Record the reading of both burettes. Repeat using 25 ml of alkali. Repeat the third time using 35 ml of alkali. Calculate each time the number of ml of the alkaline solution equivalent to one ml of the acid. Take the average of

these three determinations and calculate the grams of alkali in one ml of the unknown alkali solution (you will be told whether it is NaOH or KOH). From this value, calculate the normality of the alkaline solution.

(b) Given a standard (known) alkali solution and an unknown acid solution, titrate as indicated above, but use phenolphthalein indicator. Calculate the normality of the unknown acid solution.

Exercise No. 47

OXYCOMPOUNDS OF CHLORINE AND THEIR REACTIONS (224-228)

(a) Fill the curved portion of a test tube with KClO_3 crystals. Cover with concentrated HCl , and warm. What is the yellow gas formed in the tube? Write the equation. Pour this gas into a 100 ml beaker half filled with water, and stir. Heat the tube again, and pour the gas into the same water. Repeat until the water becomes yellow. To prevent the gas escaping into the room when you have prepared all you need, fill the tube with water and pour into the sink.

1. Test a portion of this solution, and prove that it is chlorine water.

2. Neutralize a portion with NaOH.

3. Put Mg ribbon into a portion in a test tube, and identify the gas given off.

4. Summarize the properties of this water solution. What is chlorine water? Represent chlorine water by an equilibrium equation, naming all compounds the formulas of which are employed.

Exercise No. 48

PREPARATION OF HARD BLEACH SOLUTION (225-226)

(a) Digest about 25 ml (25 g) of bleaching powder with about 100 ml of water. Stir thoroughly, allow to stand for half an hour, and filter. Is it soluble? The solution of bleaching powder is known as **hard bleach**. Test this solution for its bleaching properties by dipping colored cloth first into the bleaching solution, and then into dilute acid solution. It may be necessary to repeat this several times to remove the color completely. What is bleaching powder? Give structural formula. Can you give it a chemical name?

Exercise No. 49**PREPARATION OF SOFT BLEACH SOLUTION (225-226)**

(a) To about 50 ml of the hard bleach solution, add slowly a solution of Na_2CO_3 , a little at a time, stirring constantly. Write the formula of the precipitate formed. When no further precipitate occurs, allow to settle and decant the clear liquid. This is known as **soft bleach**. Test its bleaching properties as indicated in Exercise 48. Chemically, what is a soft bleach solution? Why is bleaching done in acid solution?

(b) Treat a portion of this bleaching solution with a solution of H_2O_2 . Explain what you observe.

Exercise No. 50**PROPERTIES OF CHLORATES AND PERCHLORATES
(227-230)**

(a) Place about one-half inch of KClO_3 crystals in a hard glass test tube. Heat and test the gas. What is it? Continue heating at as low a temperature as possible, and keep the mass just melted until practically all the gas has been given off and the mass begins to solidify. Of what does this mass consist? Write the equation.

(b) Dissolve this mass when cool, by employing about 50 ml of hot water, in about five equal portions of 10 ml each. Cool (in cold water or ice) to about 10° to 15° C., for a little while, and see if crystals form. Filter, wash with **cold** water, and dry between filter papers.

(c) Test these crystals and prove they are KClO_4 and not KClO_3 . Chlorides, chlorates, and perchlorates can be distinguished and identified by the following tests:

1. The solubility of the potassium salt increases with decrease of oxygen content.

2. Potassium perchlorate yields oxygen without melting; potassium chlorate yields oxygen and melts, and potassium chloride does not yield oxygen when heated and melts with difficulty.

3. Chlorates only are decomposed with explosive violence when a small crystal is added to warm H_2SO_4 .

4. Only solutions of chlorides acidified with HNO_3 and treated with AgNO_3 give AgCl precipitate.

5. Chlorates are reduced to chlorides by SO_2 , but perchlorates are not.

(d) Secure samples of the three types of compounds and test them by the different methods. Summarize your data indicating how these compounds may be distinguished.

All chlorates are soluble in water.

Exercise No. 51

PROPERTIES OF BROMINE AND IODINE COMPOUNDS (232-237)

(a) To a solution of KBr add one ml of CCl_4 . Cautiously, add a few drops of chlorine water (a hard bleach solution may be employed), and shake after each addition. The CCl_4 becomes reddish or yellow in color, the shade depending upon the amount of bromine liberated. Write equation.

(b) Repeat, using a solution of KI . A violet coloration of the CCl_4 indicates the presence of iodine. Now add a large excess of chlorine water. The iodine should be oxidized to iodic acid, and the solution should become colorless. Write equations representing reactions and name compounds represented by formulas used.

(c) Employing a mixture of a bromide and an iodide, show, by the addition of successive small quantities of chlorine water, that the iodine will be first liberated, then oxidized to the colorless iodic acid, and then, upon the further addition of chlorine water, the bromine is liberated. Write equations representing these reactions.

(d) Show that similar reactions occur by employing KMnO_4 after making the solution of the mixed halides slightly acid with H_2SO_4 .

(e) To 10 ml of bromine water placed in a beaker, add dilute NaOH until the color disappears. Explain by means of an equation. Write the equilibrium equation representing the system known as bromine water.

(f) To a portion of the solution of NaBrO add H_2O_2 . Write equation.

(g) To a strongly alkaline solution of NaBrO add a little urea, $\text{CO}(\text{NH}_2)_2$. What is the gas given off?

Exercise No. 52**PREPARATION AND PROPERTIES OF SULPHUR DIOXIDE
(239-242)**

(a) Place a little sulphur in a deflagrating spoon, ignite, and introduce into a dry bottle. Note the odor. Test the gas with both dry and moist litmus paper. Add water to the bottle and test the liquid with an indicator. - Write equation.

(b) Into a test tube, put some copper turnings. Add a few ml concentrated H_2SO_4 , and heat carefully. Test the escaping gas. Write equation.

(c) Repeat, using sulphur and also charcoal in place of the copper. Write system of equations representing the reaction and name all substances represented by formulas used. . Recall the relative position in the electrochemical series of H and Cu, S, and C. Compare the action of elements above hydrogen in the electrochemical series with that of those below hydrogen in the electrochemical series upon sulphuric acid.

(d) Into another test tube, put a few crystals of sodium thiosulphate and add HCl, warm, and test the gas evolved.

(e) Into a small flask, put about 5 ml (5 g) of sodium bisulphite, cover with water, insert a two-hole stopper carrying a delivery tube and a glass tube joined to a funnel by means of a rubber tube carrying a pinch clamp. Allow H_2SO_4 to flow into the flask drop by drop. (Do not have the delivery tube dipping into the liquid.) Collect several test tubes of the gas and test its color, odor, and solubility in water. Does it burn? Does it bleach?

Exercise No. 53**PROPERTIES OF SULPHUROUS ACID (239-242)**

(a) Pass some SO_2 gas into 100 ml of distilled water which has been previously boiled and cooled. What is this solution? Test this system (1) by its odor; (2) with an indicator; (3) by adding Mg ribbon and warming; (4) by adding dilute KMnO_4 solution; (5) by boiling 10 ml in an evaporating dish and testing gas given off; (6) by adding NaOH to a portion of the sulphurous acid solution until the system is neutral. Write equation. Name the salt formed.

(b) Dissolve a few ml of NaHSO_3 in a test tube of water. Test solution by (1) litmus, (2) adding HCl to a portion, warming, and testing gas evolved. Write equations involved.

Exercise No. 54

PREPARATION AND PROPERTIES OF SULPHURIC ACID, (243-251)

(a) By employing a carefully prepared solution of sulphurous acid, H_2SO_3 , which has been proved to contain no SO_4 radical (that is, no sulphate), show that it may be oxidized to sulphuric acid by oxidizing agents, while they themselves become reduced as follows:

1. In a beaker, place 5 ml of the carefully prepared solution of H_2SO_3 . Pour onto this, at the same time stirring, nitric oxide gas, NO , obtained by treating a few pieces of metallic copper with dilute 3M HNO_3 and heating. Test the resulting solution for the presence of the SO_4 radical by adding a few drops of BaCl_2 solution. Write equations representing all reactions and name all compounds the formulas of which you employed.

2. Reduce a dilute solution of FeCl_3 with the H_2SO_3 solution. Show by positive tests that the iron has been reduced and that the H_2SO_3 has been oxidized. Give all necessary equations.

3. Show the oxidizing power of chlorine water on H_2SO_3 . Name the products and write the equations.

(b) Heat very strongly some anhydrous ferrous sulphate until the residue becomes brick red and test the gases evolved at different stages. What are they? Proof? Write equations representing the reactions. Pass some of the final gas into water and test the resulting solution for SO_2 .

(c) Heat a few ml of concentrated H_2SO_4 in a crucible, **under the hood**, until copious fumes are evolved. What are they?

(d) Dilute about one ml of concentrated H_2SO_4 with two volumes of water. Should the acid be poured into the water or the water into the acid? Give your reasons for the proper method of mixing the water and concentrated H_2SO_4 . Now prepare about 10 ml of dilute H_2SO_4 of the strength stated above.

To a few ml of dilute sulphuric acid solution add a few drops of BaCl_2 solution. Stir. To this add a few ml concentrated HCl . Does the precipitate dissolve? Obtain three sulphate solutions and test as you did the dilute H_2SO_4 solution. What

are your conclusions? Is this a test for the SO_4 radical? What other salts could you use in place of the BaCl_2 ? Why? Try them. Which are the insoluble sulphates?

(e) To solutions of dilute KMnO_4 , add respectively (1) H_2S , (2) H_2SO_3 . Test the resulting solutions and prove that in all cases you have produced the SO_4 radical. How do you prove this? Explain fully all of the reactions by means of equations. Remember you do not yet know how KMnO_4 breaks down, and you just assume it yields oxygen for oxidizing purposes. Refer to your previous experiments with H_2S and with SO_2 , involving these reactions.

(f) Place a drop of concentrated H_2SO_4 on (1) a soft wood splinter, on (2) cotton, and let stand for a few minutes. Explain the action. Place a few drops of dilute H_2SO_4 on filter paper; dry on the steam bath. Explain. Concentrated H_2SO_4 can be employed for what?

Exercise No. 55

TEST FOR SULPHUR IN COMPOUNDS

How would you distinguish a **sulphite** from a **sulphate**? If the compound is insoluble in water and in dilute HCl , fuse the solid with KNaCO_3 , extract with water, and test the aqueous solution? How? Explain all reactions. Confirm this by employing some barium sulphate.

(b) How would you distinguish between a **sulphide** and a **sulphate**? If the substance is insoluble in water? Fuse some pulverized coal with NaNO_3 and Na_2CO_3 (**caution**), extract with water, filter and test the filtrate for the sulphate radical.

(c) Mix an insoluble sulphate (CaSO_4) with powdered charcoal, place it in a shallow hole bored in a piece of charcoal, and heat with the reducing portion of the blowpipe flame. Scrape the fused mass into a test tube, add about 5 ml H_2O and boil. Then place a few drops of this solution on a clean silver coin. Explain, and write equations representing all reactions.

Exercise No. 56

SULPHATES (251-258)

(a) Put a small crystal of copper sulphate into a test tube, and heat gently. Explain the result. Repeat, using sodium sulphate. Use calcium sulphate. Do you notice any difference? Explain.

(b) To 10 ml of a solution of KOH, add a few drops of phenolphthalein indicator. Now, add cautiously **very** dilute H_2SO_4 solution until the pink color just disappears. Divide this solution into two equal parts. Evaporate one portion to a small bulk, allow to stand and to crystallize. To the second portion, add a few drops of the methyl orange indicator, and then add more of the dilute H_2SO_4 , cautiously, until the solution is just acid. It is well to use a pipette, or a glass tube drawn out to a point, for the introduction of the acid. Toward the end, this must be done very carefully, using a drop at a time. Evaporate this solution to a small volume, allow to crystallize. Now pour off the liquid from the crystals in the two beakers and dry between filter paper. Examine carefully. Can you notice any difference in crystalline form? Heat some of the crystals gently in a test tube. Explain. Determine which of the two has the greater solubility in water. Explain how this is done.

Exercise No. 57

PREPARATION OF A PLASTER OF PARIS CAST (254-255)

To about 10 ml (10 g) of plaster of Paris, add enough water to make a thick paste. Put this into a small paper box made by folding a sheet of paper. While this mass is still soft, impress upon it a coin which has been thinly coated with vaseline. Remove the coin carefully, and allow the mass to harden by letting it stand until the next period. Explain. Break off a small corner of the cast, put it into a test tube, and heat it. Write equations representing all reactions.

Exercise No. 58

PROPERTIES OF ALUMINUM SULPHATE AND ALUMS (256-258)

(a) Dissolve a little alum in water, and test the solution with litmus. Do the same with ammonium sulphate crystals. Also with aluminum sulphate crystals. Explain, employing equations wherever possible.

(b) Pour into a solution of cochineal, to which a little ammonia or $\text{Ca}(\text{HCO}_3)_2$ has been added, about an equal volume of a solution of alum or of aluminum sulphate. Be sure enough NH_4OH has

been added to make the final solution alkaline. Allow to stand. Explain results. Define mordant, lake.

(c) Dissolve 50 ml (50 g) of alum in 100 ml hot water. Arrange this to stand with a string suspended in the liquid. Set aside until next period. Examine and explain. Use Cr, Fe, or Mn alum.

Exercise No. 59

PYROSULPHATES AND PERSULPHATES (258-261)

(a) Heat a few ml (g) of NaHSO_4 in a test tube until the fused mass boils quietly. Cool, extract with water, and test the solution with an indicator. Obtain some fused potassium acid sulphate. Dissolve in water, and test this solution with an indicator. Heat some in a test tube until it melts. Do the same with K_2SO_4 . How do these compounds differ?

(b) To a cold concentrated solution of KHSO_4 or NH_4HSO_4 , add H_2O_2 . Keep the solution at a temperature of about zero. Examine the crystals formed. Test by adding KI to a solution of them. Explain.

Exercise No. 60

THIOACIDS AND THIOSULPHATES (261-263)

(a) Heat a mixture of 2 parts of NaHSO_3 and one part of S in a test tube. Cool, extract with water, and filter. Treat a portion with concentrated HCl. Write equations.

(b) Dissolve about 10 ml (10 g) Na_2SO_3 in 25 ml of water, add 4 ml (4 g) of sulphur, and boil for about half an hour. Filter, and test with HCl, or set aside to crystallize.

(c) Treat a solution of $\text{Na}_2\text{S}_2\text{O}_3$ with HCl. Similarly treat Na_2SO_3 and Na_2SO_4 separately with HCl. What are the distinguishing characteristic differences? Explain how you would distinguish thiosulphates from sulphates and sulphites. Which did you prepare?

(d) Pass SO_2 into a solution of Na_2SO_3 , filter, and test the solution for a thiosulphate. How? Is a thiosulphate formed?

(e) Treat a solution of $\text{Na}_2\text{S}_2\text{O}_3$ with chlorine water. If an excess is added, introduce a little more of the thiosulphate solution. How can you tell whether you have an excess of chlorine water? Write equations.

(f) To a solution of $\text{Na}_2\text{S}_2\text{O}_3$ add a solution of iodine (prepared by adding free I to KI solution). Write equations.

(g) Prepare a little AgCl precipitate, and wash thoroughly with hot water. Treat with a solution of $\text{Na}_2\text{S}_2\text{O}_3$. Write equations. Which are the insoluble thiosulphates? Are any of these soluble in an excess of $\text{Na}_2\text{S}_2\text{O}_3$ solution?

Exercise No. 61

NITRIC OXIDE AND NITROGEN DIOXIDE (266-267)

In a test tube, place a few pieces of Cu wire or turnings. Add 5 ml of dilute HNO_3 (concentrated HNO_3 diluted with four times its volume of water) to the copper, and after inserting a stopper carrying a delivery tube, heat gently. Collect a few test tubes of the gas. By water or by air displacement? Which? Expose one tube of the gas to the air. Explain. Test this brown gas, and determine its action with a glowing splint. Take another tube of the gas and, after exposing to the air, invert over water. Explain what you observe, writing the necessary equations. Now remove, and expose the remaining gas to the air again, and explain.

Exercise No. 62

PREPARATION AND PROPERTIES OF NITRIC ACID (275-285)

(a) Place 10 ml (15 g) NaNO_3 in a retort. Cover this with concentrated H_2SO_4 and introduce the outlet end of the retort nearly to the bottom of a test tube which is immersed in cold water. Heat the retort gently until a few ml of liquid have been collected in the receiving test tube. Add a few drops of this liquid (distillate) to a half test tube of water. Test with indicator. Write equation.

(b) To copper turnings, add a few ml of concentrated HNO_3 and heat. Observe carefully the gases given off. Write system of equations.

(c) Repeat, using dilute nitric acid (about 1 part of concentrated HNO_3 to 4 parts of H_2O). Note carefully the difference in the character of the gas given off from that in (b). Write a system of equations to show this. Collect some of this gas by displacement of water. Is this gas soluble in water? On expo-

sure to the air, what happens? Expose to the air and test solubility in water of the gas formed.

(d) **Test for Nitrate Radical.**—To a test tube of water add a drop or two of dilute HNO_3 or solution of a nitrate. Mix thoroughly. Pour about half an inch of this solution into another test tube, add an equal volume of concentrated H_2SO_4 , mix thoroughly and cool under the tap. Incline the tube, and add cautiously a dilute solution of FeSO_4 so as not to mix the two solutions. Explain what occurs at the boundary where the two solutions meet. Test some other nitrate in the same manner. This is a test for the NO_3 radical.

Exercise No. 63

NITROUS OXIDE (285)

(a) Into a test tube or small flask, introduce about 10 ml (10 g) of ammonium nitrate. Insert the stopper carrying a delivery tube turned up so the gas can be collected by displacement of water. Have the water slightly warm. Why? Heat the flask very gently with a low flame. After the air in the apparatus has been expelled, collect three bottles of the gas. Cover with glass plates until used.

(b) Leave one of the bottles uncovered. Compare with nitric oxide, NO . Into one of the bottles introduce a glowing splint. Explain. Into another vessel of the gas, introduce some glowing steel wool. Write equations illustrating all of these chemical changes.

Exercise No. 64

NITROUS ACID AND ITS SALTS (283-285)

(a) Mix some sodium nitrate 5 ml (5 g) and metallic lead 2 ml (5 g) in an iron crucible and heat, stirring frequently to mix thoroughly. When practically all of the lead has disappeared, cool, cover with water, heat to boiling, allow to cool, and filter. Treat the remainder in the crucible again with boiling water, and add the two filtrates together. Do not use more than 50 ml of water for each extraction.

(b) To a portion of this solution, add a few drops of H_2SO_4 . Test the solution by adding a few drops of it to a starch-KI

solution. Explain your results. Compare your results with the action of H_2SO_4 on sodium nitrate.

(c) Heat separately $\text{Pb}(\text{NO}_3)_2$, $\text{Cu}(\text{NO}_3)_2$, NaNO_3 , NaNO_2 . What are the differences?

(d) Repeat (d) of Exercise No. 62, using HNO_2 or a nitrite. Result? What are the distinguishing tests for nitrous and nitric acid radicals?

Exercise No. 65

ORTHOPHOSPHORIC ACID AND ORTHOPHOSPHATES (294-298)

(a) To distilled water, add a little P_2O_5 (**caution**). Test with litmus. Result? Add about 1 ml of this solution to 5 ml of water and to this add a drop or two of AgNO_3 solution. Result? Color of precipitate? Write an equation and name all compounds whose formulas were used.

(b) To another portion of the solution, add a drop or two of HNO_3 , and boil for several minutes. Add an equal volume of this solution to 5 ml of water and to this add a few drops of AgNO_3 solution. Carefully neutralize this with a dilute solution of NH_4OH . Result? Explain. Is this result the same as that above? Represent by equations, and name all substances the formulas of which were used.

(c) To a dilute solution of Na_2HPO_4 add a few ml of magnesia mixture. (This is prepared by adding to a MgSO_4 solution sufficient NH_4Cl to prevent the formation of a precipitate when made alkaline with NH_4OH .) Write equation assuming the precipitate is MgNH_4PO_4 .

(d) Acidify a solution of Na_2HPO_4 with HNO_3 , warm, and add an equal volume of ammonium molybdate solution. You cannot write the equation. Under properly regulated conditions, the precipitate is ammonium phosphomolybdate $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3$. This is one of the most characteristic tests of orthophosphates.

Exercise No. 66

SODIUM METAPHOSPHATE BEAD (297)

(a) Prepare a sodium metaphosphate bead by dipping the hot loop of a platinum wire into pulverized $\text{NH}_4\text{NaHPO}_4$ (microcosmic salt) and heat in the flame until it is clear. Write equation

representing the reaction and the compound formed. Name? Now touch the hot bead to a solid compound containing Mn, and heat again in the flame until the bead is transparent. Repeat until sufficient intensity of color has been obtained.

(b) Repeat, using compounds of Co, Ni, and Cu.

Exercise No. 67

THE AMPHOTERIC CHARACTER OF METALLIC ELEMENTS

(a) 1. To a few pieces of Al, add 10 ml dilute HCl. Warm gently, to facilitate the reaction. Record your observations. What gas is given off? To another portion of Al, add NaOH solution. Record your observations. What gas is given off? Write equations showing the reaction with both HCl and NaOH. Name all substances the formulas of which were used.

2. To a few pieces of granulated zinc (about 30 mesh), add 10 ml dilute HCl. Record your observations. To other pieces of Zn, add NaOH solution, and warm. Record observations. Write equations in both cases, and name all substances represented by the formulas used. What is the valence of Al and of Zn in the different compounds? What do you understand by amphoteric character?

(b) Pour off the liquid from the unused Al which has been treated with HCl, and dilute to about 25 ml with water.

1. To about half of this, add small amounts of NaOH solution, a little at a time, until the solution is just alkaline, and a permanent precipitate is formed. What is the precipitate? Stir the mixture thoroughly, and pour out enough to cover the bottom of a small beaker. To this, add (**cautiously**) the NaOH solution until the precipitate disappears. Write the equation representing this reaction, naming all of the substances.

2. Transfer another portion of this precipitate to another beaker, and add HCl solution cautiously until the precipitate disappears. Write the equation naming all of the substances. What has happened in each case? Explain why this shows the amphoteric character of aluminum. Write equations representing all the chemical reactions, naming all substances the formula of which you used.

(c) Pour off the liquid from the unused zinc which has been treated with HCl, dilute to about 25 ml and pour about half into a small beaker. Add enough NaOH solution to just neutralize

the acid and produce a permanent precipitate. Write an equation illustrating the reaction, and name all substances represented by the formulas. Shake or stir to uniformly distribute the precipitate, and pour out a few milliliters into each of two test tubes. To contents of one test tube, add an excess of the NaOH solution and to the other, an excess of HCl. Explain what happened, and why the precipitate disappeared in both cases. Write equations. Write the names of all substances represented, and state why these reactions show the amphoteric character of zinc.

(d) To 5 ml of a solution of MnSO_4 , in which the Mn acts as a basic element, add an equal volume of water and then sufficient NaOH solution so that on further addition of NaOH no further precipitation takes place. Warm, and allow to stand so that the precipitate will settle. If the precipitate is thick and gelatinous, a little water should be added so that the precipitate will settle more readily. Filter a portion of this, wash the precipitate, and expose it to the air. Describe and explain. Write the chemical equation, and name all of the substances the formulas of which you have used.

(e) 1. To about 5 ml of a solution of potassium permanganate, KMnO_4 , add an equal volume of water and then a few drops of the NaOH solution. Assure yourself that a reaction does or does not take place. Explain the difference in the action of the manganese in this and the previous case. How would you recognize manganese functioning as a metal, that is, as a basic element?

2. Dilute a few ml of KMnO_4 solution with an equal volume of water, and add a solution of H_2SO_3 (sulphurous acid) drop by drop, until, with frequent stirring, the color disappears. Test the colorless solution for manganese as a basic element. What is the valence of Mn when it functions in each of these two ways? What have you done to the Mn by means of H_2SO_3 ? What kind of a reagent is the latter?

3. Hydrogen peroxide, H_2O_2 , could be used in place of H_2SO_3 . Try it. What kind of a reagent is KMnO_4 ?

4. Summarize the manner in which you can show the dual character of manganese in its compounds?

OPTIONAL

(f) Dilute 5 ml of chromium sulphate $\text{Cr}_2(\text{SO}_4)_3$ solution with an equal volume of water, and add a solution of NaOH. Employ 5 ml of a solution of potassium chromate (K_2CrO_4), dilute, and add

NaOH solution. Explain the difference, with the aid of the necessary equations, naming all compounds the formulas of which you have employed, and show that chromium is amphoteric.

(g) 1. Dilute 5 ml of the potassium chromate solution with an equal volume of water, add about 5 ml of H_2SO_3 solution, and heat. Notice any change in color. Make the solution alkaline with NaOH. Is there a precipitate formed? What is it? What does this indicate?¹

2. Transfer some of this precipitate, which has been washed by decantation, to a small beaker, cover with water and then add a small portion of sodium peroxide. Heat, notice the change in color, and then add a few drops of NaOH solution. Is a precipitate formed? What does this show? Explain what you have done to the chromium during these reactions. What are the valences of chromium? What is the valence of chromium when it acts as a basic element? An acidic element? How would you classify the above changes? Contrast characteristics of $\text{Cr}_2(\text{SO}_4)_3$ and K_2CrO_4 by tabulating their properties.

Exercise No. 68

PROPERTIES OF MANGANESE DIOXIDE (366)

Treat small portions of MnO_2 with (1) dilute H_2SO_4 , and (2) concentrated HCl. Write equations illustrating the reactions in each case, employing the system-of-equations method. What gas is given off in each case? How did you prove it?

Exercise No. 69

PREPARATION OF MANGANATES BY FUSION (366-368)

(a) Place a little Na_2O_2 in a deflagrating spoon, cover with MnO_2 , and add enough additional Na_2O_2 to cover, thus making the spoon about two-thirds full. Heat cautiously until effervescence ceases, and the mass is bluish green in color. Cool, dissolve in water, allow to stand, and decant the clear supernatant green

¹In place of H_2SO_3 , ethyl alcohol could be used. In this case, the chromate solution has to be acidified with HCl, the alcohol added, and then the system heated for some time, to produce the reaction. The solution turns green, and an aldehyde with a very characteristic odor is evolved.

solution. Write a system of equations to illustrate the reaction. To what is the green color due?

1. Add a few drops of this green solution to about 50 ml H_2O . Note change in color. If solution is still green, pass CO_2 through one portion of it, and add HCl to another portion. Explain change in color by a system-of-equations method.

2. Add about 5 ml of the original solution obtained from the fusion to 50 ml of water. Acidify with H_2SO_4 . Divide into three parts. To one portion, add H_2S , to another, add H_2SO_3 , and to the third, add H_2O_2 or chlorine water. What happens in each case? Write equation representing the reduction of the pink solution.

(b) Add a few ml of the original manganate solution to 50 ml H_2O . Acidify with H_2SO_4 , and divide into two parts. To one portion, add a solution of FeSO_4 , and to the other, add a solution of oxalic acid $(\text{COOH})_2$. Warm these, and explain the change in color. Write equations, employing system-of-equations method.

Exercise No. 70

TESTS FOR MANGANESE IN COMPOUNDS

To a solution of MnSO_4 add enough NaOH to make the solution alkaline. Heat and filter. Wash the precipitate.

(a) Make a borax bead, by dipping the red hot looped end of a platinum test wire into powdered borax and heating. Repeat this process until a transparent borax bead is obtained. Heat again, and dip into the precipitate of manganese. Heat again until the bead clears. Repeat, dipping the bead into the compound to be tested, and reheating, until a highly-colored clear bead is obtained. Avoid, however, making the bead sufficiently highly-colored to obscure the characteristic color. If it becomes too intense, break it into pieces, and introduce a small portion into a new borax bead. The amethyst color is characteristic of manganese.

(b) Place a portion of the precipitate $\text{Mn}(\text{OH})_2$ into a test tube, cover it with HNO_3 , add a few ml (grams) of lead dioxide (or red lead), and heat. Dilute to the top of the test tube with water, and allow to stand. Explain why the solution is pink. PbO_2 is a strong oxidizing agent.

Exercise No. 71**PREPARATION OF CHROMIUM TRIOXIDE (370)**

To 10 ml of a concentrated solution of $\text{Na}_2\text{Cr}_2\text{O}_7$ (50 ml (75 g) in 30 ml of H_2O), add 15 ml concentrated H_2SO_4 . Cool, and allow to stand until crystals separate and collect at the bottom of the beaker. Decant as closely as possible, invert the vessel, and allow to drain thoroughly. Remove some crystals, place them on a porous plate, and let them dry. Test their solubility. Test the solution with litmus. Equations. What are the red crystals?

Exercise No. 72**PROPERTIES OF CHROMIUM COMPOUNDS (369-371)**

(a) To a solution of $\text{Cr}_2(\text{SO}_4)_3$, add NH_4OH until the solution is just alkaline. Heat, filter, and wash the precipitate thoroughly. Write the equation. If this precipitate were ignited, what would be formed?

(b) Place a portion of the precipitate from the preceding in a beaker, cover with water, and add cautiously, a little at a time, about half a teaspoonful of Na_2O_2 . Heat to drive off the excess oxygen, dilute with water. What is formed? Write the equation to represent the reaction.

1. Test the solution. Dilute with an equal volume of water and pour 5 ml into each of three test tubes. Acidify one portion with acetic acid, and add lead acetate; acidify another portion with HCl and add a solution of BaCl_2 ; acidify a third portion with HNO_3 and add AgNO_3 . Write the system of equations representing reactions between the chromate and the precipitating reagents.

2. What would happen if the acids HCl and HNO_3 were interchanged and the same precipitating reagents used? Try this. Write equations representing all the reactions, and name the substances the formulas of which you have used.

Exercise No. 73**PREPARATION OF CHROMATES AND THEIR REDUCTION (371-372)**

(a) Mix about equal portions of solid Na_2CO_3 and solid NaNO_3 , and add to this about one-fifth their volume of the precipitated $\text{Cr}(\text{OH})_3$ from Exercise 72, and fuse on a porcelain cru-

cible cover. This is analogous to the Mn experiment in Exercise 69. Write equations, and compare them with the Mn reaction. Dissolve the fused mass, dilute, and test as in the preceding exercise.

(b) To about 2 ml concentrated HCl in a beaker, add 10 ml of your chromate solution, and dilute to about 50 ml. Add 2 ml ethyl alcohol, and warm. Continue heating until a dark green solution is obtained. To what is this color due? To what is the change due? Write equations showing this, employing the system-of-equations method.

(c) Acidify 10 ml of distilled water in a test tube with H_2SO_4 , add a few ml of H_2O_2 , and 2 ml of ether. Introduce a few drops of the chromate solution to be tested. Observe the color. Shake thoroughly, allow to settle, and record your observations.

PART II
ORGANIC CHEMISTRY EXERCISES

Exercise No. 74

PREPARATION OF METHANE (425-432)

(a) Grind together intimately in a mortar 4 grams of **fused** sodium acetate (CH_3COONa), and 8 grams of powdered soda-lime ($\text{CaO} + \text{NaOH}$). Transfer to a hard glass test tube, insert a rubber stopper carrying a glass delivery tube so arranged as to collect the gas evolved by displacement of water. Support the test tube so that the outlet is lower than the closed end which is to be heated. Heat it cautiously by keeping the low flame in motion, and after the air has been expelled and there is a steady flow of gas, collect several test tubes of the gas.

(b) Ascertain the properties of the gas, including odor, color, specific gravity with respect to air, solubility, inflammability. What are the products formed when it burns?

(c) Mix equal volumes of the gas and air by filling a test tube half full of the gas and then replacing the remainder of the water with air. Wrap the test tube in a towel and cautiously bring the mixture over the flame. Result?

(d) What results when the gas is treated with a very dilute solution of KMnO_4 ?

Exercise No. 75

PREPARATION OF ETHYLENE (433)

(a) To 10 ml of water in a beaker, add 30 ml of H_2SO_4 (conc). Cool under the tap. In a 100 ml flask place a little clean sand, and pour in 10 ml of ethyl alcohol. Now pour in the **cold** acid and mix thoroughly by shaking. Insert the stopper carrying a delivery tube for collecting the gas by displacement of water.

(b) Heat gently on a wire gauze, and avoid too violent evolution of gas. Collect several test tubes of the gas. Remove delivery tube from the water, and put out flame. Fill flask with water, allow sand to settle, and pour off the liquid into the sink. Fill flask with water, allow to settle, decant, and finally put sand into slop jar.

1. Note color, specific gravity with respect to air, solubility, inflammability of gas, and color of the flame. Note particularly luminosity of flame, and compare with that of methane.

2. Mix equal volumes of the gas and air, and drop a lighted match into the test tube, which must stand in a bottle for protection. Explain.

3. To one test tube of gas, add a few ml of dilute KMnO_4 solution. Explain and compare with methane.

4. Add a few drops of bromine water to a test tube of the gas. Explain. Are any oily drops visible? Test gas above liquid with moist litmus, and then test the liquid.

5. Collect a bottle of illuminating gas and test by methods employed above. Which of these gases do you think is present in the illuminating gas? Give your evidence.

Exercise No. 76

PREPARATION OF ACETYLENE (434-435)

(a) Into a small flask (100 ml), pour about 50 ml of water. Drop into this a small piece (the size of a pea) of CaC_2 , and immediately insert the stopper carrying a delivery tube. Collect several test tubes of the gas after the air has been displaced from the apparatus. Ignite the gas at the end of the delivery tube, and compare its luminosity with that of methane and of ethylene.

1. To the gas in one test tube, add about 1 ml of bromine water. Shake. Result? Compare with action on methane and ethylene.

2. Add a few milliliters of dilute KMnO_4 solution to a tube of gas. Result?

(b) Tabulate all the properties of methane, ethylene, and acetylene, and compare the distinguishing characteristics.

Exercise No. 77

PROPERTIES OF GASOLINE AND OF KEROSENE (441-447)

(a) Determine the specific gravity of the samples of gasoline and of kerosene assigned you, by means of a hydrometer.

(b) Place about 2 ml of kerosene in a test tube, and add 5 ml HNO_3 . Repeat, using H_2SO_4 (concentrated) and using NaOH (dilute solution). Is heat evolved, or is there, in each case, any

change in color, or evidence of a reaction? Record your observations and conclusions.

(c) Repeat, using gasoline.

(d) Place 10 ml of dry high test gasoline in a dry test tube, and add 2 ml of a 5 per cent solution of Br in CCl_4 . Pour one-half of this into a dry test tube, and set away in your desk in the dark. Place the other portion in direct sunlight. Observe in 10 minutes. Test with moist litmus paper, also by blowing your breath across the top of the test tube. Explain the differences.

(e) **Flash Point.**—1. Pass a small flame over a few drops of gasoline in a watch glass. Result? Explain. Repeat, using kerosene. Insert a burning match into the kerosene on the watch glass. Explain.

2. Pour kerosene into a small 50 ml beaker until it is nearly full. Support this in a beaker of water, by means of a wire or a piece of pasteboard in which the beaker is fitted snugly. Support the whole on a wire gauze above a flame, and have a thermometer suspended in the kerosene. Provide an asbestos sheet, or board, with which to smother the flame when the kerosene takes fire. Provide a small flame, about one-fourth inch long, by connecting your blow pipe to the gas supply. Now heat the beaker containing the water, and when the temperature of the kerosene is about 50° , pass the small flame across the surface of the kerosene but do not touch it. Continue to heat the system, and test for the vapor above the surface of the kerosene for every degree rise. Record the temperature at which the vapor from the kerosene takes fire (ignites). The vapor flashes when it ignites and is many times difficult to see. Extinguish all flames as soon as the flash point is determined. If no flash point is detected when the temperature reaches 95° , consult the instructor.

Set the beaker containing the kerosene in a beaker of cold water, and when it is cool, return it to the bottle provided for this purpose and marked **Refuse Kerosene from the Flash Point Determinations.**¹

3. Summarize all your results and show in what ways gasoline differs from kerosene.

¹ New York Oil Tester to be employed, if available.

Exercise No. 78

PROPERTIES OF AND TESTS FOR METHANOL (458-463)

(a) To about 3 ml of water, add 5 ml methanol. Into this, dip a hot copper wire coil obtained by winding a few turns of copper wire around a lead pencil or glass tube, leaving enough of the wire to serve as a handle. Heat this coil in a flame until red, and insert it in the solution of methanol contained in a test tube immersed in a beaker of water. Repeat this about five times. Note, each time, the odor of the solution after removing the wire. What is the color of the copper wire when it is removed from the flame and cooled? What is the color when it is removed from the methanol solution? How do you account for this?

To a portion of the solution, add about two drops of a 0.5 per cent solution of resorcin. Shake. Pour this carefully, so as not to mix, onto the surface of about 3 ml of concentrated H_2SO_4 in a test tube. The violet colored ring at the surface of the liquids is the characteristic test for formaldehyde, HCHO :

(b) In a test tube, place 1 ml H_2SO_4 , and about 0.2 g of salicylic acid. Introduce into this 1 ml of methanol and heat. Observe the characteristic odor of methyl salicylate. What is its common name?

Exercise No. 79

PROPERTIES AND TEST FOR ETHANOL (463-468)

(a) **Oxidation of Ethanol.**—Refer to Exercise No. 73, and repeat by heating about 5 ml of $\text{K}_2\text{Cr}_2\text{O}_7$ solution to which has been added 1 ml of ethanol and 5 ml H_2SO_4 (dilute). Note the change in color, and the characteristic odor of the volatile product, acetaldehyde, CH_3CHO . Write the system of equations representing the chemical reactions. To what is the green color of the resulting solution due?

(b) **Ethyl Acetate Test.**—In a test tube, place about 1 ml of $\text{C}_2\text{H}_5\text{OH}$. Add 1 ml of glacial acetic acid, CH_3COOH , and then add 2 ml H_2SO_4 (concentrated). Warm gently, and note the odor of the volatilized product. Write equation representing the reaction.

(c) **Iodoform Test.**—In a test tube, place 5 ml H_2O , and add 1 ml of a 10 per cent solution of NaOH . Now, add 5 drops of ethanol, then a solution of iodine in KI , drop by drop, until a

faint yellow color appears. Warm gently until the tube feels warm to the hand. Allow to stand for a few minutes. Observe the color and odor of the precipitate, which is iodoform CHI_3 .

Exercise No. 80

PROPERTIES OF GLYCEROL (472-473)

(a) Test the solubility of glycerol in water, ethanol, benzene, and ether.

(b) **Acrolein.**—Into a test tube, introduce enough powdered KHSO_4 to fill the curved portion. Add about 5 drops of glycerol, and heat strongly. Note the odor of the acrolein. Have you noticed this odor about the kitchen at home?

(c) **Borax Bead Test.**—Prepare a borax bead, and introduce it into an aqueous solution of glycerol. Heat in the flame, and note the color of the flame.

Exercise No. 81

MOLISCH REACTION FOR CARBOHYDRATES (486-488)

Moisten with 10 drops of water, a small fragment of the carbohydrate to be tested and then add 2 drops of a 10 per cent solution of α -naphthol in chloroform (or alcohol). Now pour 1 to 2 ml concentrated H_2SO_4 down the side of the test tube so as to underlay the material being tested. Watch for the appearance of a colored ring at the intersection, which changes to a violet color if a carbohydrate is present. Shake, and after 1-2 minutes, dilute with 5 ml of water, and look for the appearance of a violet colored precipitate. Add NaOH solution, and note that the system becomes colorless.

Test samples of the following: starch, filter paper, sugar, gum arabic.

Exercise No. 82

SOME PROPERTIES AND TESTS FOR DIFFERENT SUGARS (488-489)

Secure a sample of the following sugars: dextrose (glucose), maltose, levulose, lactose, and sucrose.

Perform the following tests on each sample, and then tabulate your results, indicating how you can distinguish the sugars:

(a) Taste each sugar, using equal quantities. Indicate the relative degree of sweetness.

(b) Test their solubility in water, alcohol, and ether.

(c) Determine whether a mirror is formed when a dilute solution of one of the above sugars is added to a dilute ammoniacal solution of AgNO_3 . To a solution of AgNO_3 add enough NH_4OH to just dissolve the precipitate first formed upon the addition of the NH_4OH . Add equal volume of a 10 per cent solution of NaOH in a test tube cleaned with hot NaOH solution and washed with distilled water. Add a few drops of the solution to be tested. Set aside, and look for the appearance of the metallic silver, which should be deposited in the form of a mirror. This test should be made in the cold. It is known as Tollen's reaction.

(d) **Reduction of Fehling's Solution.**—To 3 ml of the CuSO_4 solution (A) add 3 ml of the alkaline tartrate solution (B). Then add to this about 5 ml of a dilute sugar solution to be tested. Heat the solution to boiling, and ascertain whether there is a red precipitate of Cu_2O formed.

(e) **Fermentation of Sugars.**—To about 10 ml of a 10 per cent solution of each of the sugars in separate test tubes, add about one-quarter of a yeast cake which has been rubbed up together with 10 ml of water in a mortar. Put cotton in the tubes, and allow to remain until the next period. Determine which sugars have undergone fermentation.

Exercise No. 83

FORMATION OF CARAMEL

Put about one-half inch of cane sugar into a test tube and heat gently, high above the flame, until there is a marked change in color. Taste this product and test its solubility in water.

Exercise No. 84

INVERSION OF SUCROSE (489-490)

To about 10 ml of a 2 per cent solution of cane sugar, add 1 ml of concentrated HCl and boil for 5 minutes. Neutralize the acid with a little NaOH solution and test for a reducing sugar by means of Fehling's solution.

Exercise No. 85**DETERMINATION OF THE PRESENCE OF REDUCING SUGAR IN MILK**

To about 25 ml of milk, add dilute acetic acid as long as a precipitate forms. Stir to collect the precipitated casein, and filter. Neutralize with dilute NaOH, and test a portion of this with Fehling's solution for reducing sugars. Which one is present in milk?

Exercise No. 86**PREPARATION OF STARCH PASTE AND TESTS FOR STARCH (493-494)**

(a) Grind about a gram of starch in a mortar with about 10 ml of water. Pour this into about 250 ml of boiling water, and stir continuously. Heat for a few minutes, set aside, and allow to cool. This is starch paste.

(b) Add a few drops of this starch paste to 100 ml of water. What is the resulting system? Add to this, one drop of KI solution. Result? Now add a few drops of freshly prepared chlorine water, and explain what takes place. Add chlorine water in excess. What is the effect on the color?

(c) Taking separate portions, try the effect of the addition of NaOH solution to one of the blue solutions, H_2SO_3 to another, H_2S , and $\text{Na}_2\text{S}_2\text{O}_3$ to other separate portions. What results are observed and what are your conclusions in each case? Does heat destroy the blue color? Does it reappear in cooling? Is this a delicate test for starch? Can it be used as a test for iodine?

(d) Stir some starch in cold water, let it settle, and test the supernatant liquid for starch. Test your filter paper for starch. Test a new piece of towelling. Test some baking-powder for starch.

Exercise No. 87**CONVERSION OF STARCH TO REDUCING SUGARS (494-495)**

(a) Test the starch paste made above with Fehling's solution.

(b) To about 50 ml of starch paste, add 50 ml of concentrated HCl, and heat to boiling in a flask into the neck of which is put a funnel. Boil for about half an hour. Remove some of

the liquid, and test it for starch. Also test another portion with Fehling's solution. Boil again for about half an hour, and test again for starch. Explain and write equation to represent the reaction.

(c) Grind a piece of cracker in a mortar with a little water and test the liquid with Fehling's solution. Result?

(d) Chew a portion of a cracker for a few minutes and then grind this mass with a little water and test with Fehling's solution. What does the saliva contain that makes the difference? What is the importance of this reaction? Why chew starchy foods thoroughly.

Exercise No. 88

SOLUBILITY OF CELLULOSE IN SCHWEITZER'S REAGENT¹

To about 5 ml of the reagent, add pieces of filter paper (or cotton), then neutralize the solution by pouring it into dilute HCl. What is the character of the precipitate?

Exercise No. 89

PREPARATION OF PARCHMENT PAPER

Mix 25 ml concentrated H_2SO_4 and 15 ml H_2O . (Care) Cool to 15°C . Immerse strips of dry filter paper for 15 to 20 seconds in this acid solution. Remove the paper, wash in running water and then in dilute NH_4OH , and finally in water. What is the appearance of this paper? Is it tougher than the untreated filter paper?

Exercise No. 90

ANALYSIS OF TEXTILE FIBERS OR CLOTH

I. The material is a mixture of silk, wool, and cotton.— Usually it is necessary to remove the sizing materials or dressings in the cloth if it is new material. This is done by boiling about

¹ Schweitzer's Reagent is prepared by dissolving 5 grams of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 100 ml of water. Add NaOH as long as a precipitate is formed. Allow to settle, wash by decantation with 500 ml of water and filter through cotton, washing until free from sulphates. Dissolve in 10 ml concentrated NH_4OH .

2–5 grams of the material in 100 ml of 0.5 per cent HCl solution for about 10 minutes. Extract with alcohol and ether, and dry.

(a) *Silk*.—Add about 60 ml of basic zinc chloride solution to the material, and heat to boiling. Remove the heat and stir, allow to settle, decant, and add 50 ml of water and filter. Wash with water. If the material is pure natural silk, no residue will be left. Any residue is wool and cotton.

The zinc chloride solution is made as follows: Add an excess of Zn metal to concentrated HCl and let stand 24 hours. Filter through glass wool, and acidify slightly with HCl.

(b) *Wool*.—The residue from (a) is treated with about 100 ml of a 5 per cent solution of KOH and boiled gently for about 15 minutes. Pour into about 500 ml of water, allow the fiber to settle, and filter by decantation. Transfer the fiber to a filter paper, and wash with dilute acetic acid. The residue is cotton or some other vegetable fibers.

II. The material is a cotton-wool mixture.—The sizing material is removed by boiling in about 100 ml of a 0.5 per cent HCl solution for 15 minutes. Filter, and wash thoroughly.

(a) *To prove which fiber is present in the greater quantity.*—Into about 100 ml of a 0.3 per cent solution of indigo carmine containing 0.2 per cent of acetic acid, place 10 grams of the material to be analyzed. Have the liquid at the boiling temperature when the fabric is added, and maintain at this temperature for about 15 minutes, working the goods thoroughly. The cotton will be dyed a pale blue, while the wool will be dyed a much deeper blue. By means of a hand lens, the approximate quantities of the wool and cotton can be readily estimated.

(b) *If the goods are high in wool*, boil the material in a 5 per cent KOH solution for about 20 minutes, and proceed as stated above in I b.

(c) *If the goods are high in cotton*, place about 10 grams of the material, after the dressing has been removed, in 200 ml of a cool (room temperature) solution of sulphuric acid made by mixing 25 ml of water and 80 ml of concentrated H_2SO_4 . Work the material in the solution for about 15 minutes, removing it frequently to another beaker of fresh water to remove the slimy material formed on the cotton by the action of the H_2SO_4 . Finally wash by decantation, and filter. The residue will be wool. From the residues from (b) and (c), the relative quantities of wool and cotton in the sample can be roughly estimated.

(d) *Rayon*.—Rayon of all varieties is dissolved by a cold solution of chromic acid. Real silk dissolves slowly, cotton and other vegetable fibers are not affected.

By Loew's reagent, real silk is dissolved immediately. It does not affect rayon, cotton, and wool. Loew's reagent is prepared by dissolving 16 grams of copper sulphate in 150 ml of water and adding 10 grams of glycerol. Sodium hydroxide is then added until the precipitate that first forms just dissolves, and a homogeneous system is obtained.

Obtain samples of various materials and determine what the constituents are.

Exercise No. 91

PREPARATION OF ACETIC ACID (546-547)

Into a hard glass test tube introduce about one inch of sodium acetate. Onto this pour about 5 ml of a solution of H_2SO_4 obtained by mixing equal volumes of concentrated acid and water. (Care). Insert the cork, putting through it a delivery tube bent so that it will come just to the surface of 10 ml of water in a small beaker. Heat the tube and its contents cautiously to boiling, and until some liquid has distilled over. Test the water in the beaker for an acid reaction. Write equations representing all reactions.

Exercise No. 92

ANALYSIS OF VINEGAR

Test a sample of vinegar for acid. How would you determine the exact quantity of acid in a sample of vinegar? Refer to Exercise 46 and write out your method in detail. Obtain from the instructor a sample of vinegar and the necessary apparatus, including standard solutions, and analyze the vinegar.

Exercise No. 93

PREPARATION OF SOAP (573-575)

(a) To about 10 ml of an approximately 50 per cent aqueous solution of NaOH , add about 25 grams of lard or of cotton seed oil in a 150 ml beaker. Suspend this in a 250 ml beaker containing

boiling water, and heat cautiously, frequently stirring, for about 45 to 60 minutes. Cool.

Explain the reactions by means of equations, assuming that the fat or oil contains stearin, $C_3H_5(C_{17}H_{35}COO)_3$.

1. To half of the soap solution, add slowly, with frequent stirring, an equal volume of a saturated salt solution. Filter off the precipitate, and wash with 50 ml saturated salt solution. Drain and allow to dry. Test for free alkali by adding a drop of phenolphthalein to the dry soap. From what has the soap been separated by this **salting out process**?

2. Cut into shavings about half of the soap you prepared, add to 200 ml of water, and heat until dissolved.

In a separate test tube, place a solution of (1) $CaSO_4$, (2) $MgSO_4$, (3) $Ca(HCO_3)_2$, and add to each a little of the soap solution. Explain the reactions by equations, assuming the fat or oil to contain stearin.

3. Take the remainder of the soap solution from (a) 1, and add enough dilute H_2SO_4 or HCl to make the solution acid. A precipitate is formed. Heat until there is formed a clear layer of oil on top of the water layer. Be careful not to boil the solution, as the liquid may spatter out. Cool in cold water, and remove the solid layer from the top. This contains the solid fatty acids, and may consist of a mixture of a number of them. Assuming one of them, show by equations how it was obtained.

(b) Place about 10 ml of an alcoholic solution of KOH (40 grams per liter) in an Erlenmeyer flask (100 ml) and then introduce about 10 drops of cottonseed oil, olive oil, corn oil, or any salad oil, or an equivalent quantity of lard. Heat this on a water bath (a beaker of water may be used) until the system appears homogeneous. Add about 75 ml H_2O and dissolve the syrupy mass. Write equations illustrating all the reactions, assuming that the oil or fat contains olein, $C_3H_5(C_{17}H_{33}COO)_3$, palmitin, $C_3H_5(C_{15}H_{31}COO)_3$, or butyrin, $C_3H_5(C_3H_7COO)_3$.

Exercise No. 94

ANALYSIS OF SOAP

Weigh out accurately 5 grams of the soap designated. Dissolve in 200 ml of water, and then add 2 ml of methyl orange indicator. Now titrate this accurately with the standard acid

solution provided for this purpose. (Ask the instructor for one.) From the number of milliliters of the acid used, the strength of which is given, calculate the grams of NaOH equivalent to this. From this, calculate what percentage of the original soap is sodium oxide. This expresses the **total alkali** in the soap. (See Exercise 46.)

Assuming the soap is sodium stearate, calculate the percentage of stearic acid in the soap.

PART III
QUALITATIVE ANALYSIS

QUALITATIVE ANALYSIS

Chemical analysis is performed to determine (a) the elements, radicals, and compounds of which a system is composed (qualitative analysis), and (b) the relative weights of each of these in the system (quantitative analysis).

The system to be analyzed may be a solid, a liquid, a gas, or any mixture of these. In general, a qualitative analysis of a system consists in the separation of the system into parts through chemical reaction, by which new compounds are formed containing different parts, and the identification of these by characteristic properties such as solubility and color reactions.

In this elementary work in qualitative analysis, the procedures are designed only for the analysis of the simpler types of systems which contain the more common elements in the less complicated combinations. If tests show that organic matter, phosphates, silicates, or borates are in a system, a more extended scheme of analysis than that given in this book must be used.

In general, it is not practical to use the whole of a system in making an analysis of it. A small portion called a sample is usually taken to work with. Great care should be exercised that the sample is representative of the system. After the sample is secured, it should be protected so that it does not change before it is examined.

Remember that an unknown system may be poisonous or corrosive; consequently, be very careful in tasting, smelling, and touching.

A sample may be contaminated while you are working with it. To prevent this, work only with clean apparatus and reagents of known purity. Never touch a sample directly with your fingers. Use only distilled water. Protect the sample from the air of the laboratory as much as possible.

Before starting work, have a notebook ready to record each bit of evidence secured. Keep notes very carefully and systematically, so that a ready reference to previous work is always available. **Record everything observed directly in the notebook at the time of observation.**

I. BASIC CONSTITUENTS

A. Preliminary Classification into Groups

A systematic procedure for the separation and identification of the basic constituents consists in separating the metals into groups by the addition of reagents designated "group reagents" and then further separating, and identifying by special reagents which produce characteristic precipitates or color reactions. The groupings are represented in the table on the left, in which is also indicated the valence of the metal.

QUALITATIVE
GROUP
ARRANGEMENT
OF THE COM-
MON METALS

Pb^{II}

Hg^I

Ag^I

Pb^{II}

Bi^{III}

Cu^{II}

Cd^{II}

Hg^{II}

As^{III}

As^V

As^VO₄

Sb^{III}

Sn^{II}

Sn^{IV}

Ni^{II}

Co^{II}

Zn^{II}

Mn^{II}

Mn^{VII}O₄

Fe^{II}

Fe^{III}

Al^{III}

Cr^{III}

Cr^{VI}O₇

Ba^{II}

Sr^{II}

Ca^{II}

Mg^{II}

K^I

Na^I

NH₄^I

If a clear water solution is assumed to contain soluble salts of the metals listed in this table and HCl is added to it in slight excess, a precipitate will be formed which consists of PbCl₂, HgCl, and AgCl. These metals precipitated by HCl constitute Group I which consists of Pb^{II}, Hg^I, and Ag^I. Their chlorides are insoluble in dilute acid. The precipitating agent is HCl. The insoluble chlorides are separated by filtration, and are in turn separated and identified by the procedure given under Group I, page 93.

The filtrate may contain the remainder of the basic constituents listed in the table above, as well as the radicals AsO₄, MnO₄, and Cr₂O₇. If this filtrate, acid with HCl, is saturated with H₂S and heated, there will be precipitated sulphides of the second group of metals given in the table, and compounds containing the radicals AsO₄, MnO₄, and Cr₂O₇, will be reduced to compounds containing respectively As^{III}, Mn^{II} and Cr^{III}. The compounds containing Fe^{III} will be reduced to compounds containing Fe^{II}. This division is known as Group II, and comprises Pb^{II}, Bi^{III}, Cu^{II}, Cd^{II}, Hg^{II}, As^{III}, Sb^{III} and Sn^{II}.

The sulphides of these are precipitated in acid solution of the proper concentration by H_2S . The Group II reagent is H_2S with HCl . The members of this group are separated and identified according to the procedure given for Group II, page 94.

The filtrate from Group II may contain the remainder of the basic constituents listed in the table on page 91. If this is made alkaline with NH_4OH , NH_4Cl added to prevent the precipitation of $\text{Mg}(\text{OH})_2$, and H_2S added, the precipitate consists of NiS , CoS , ZnS , MnS , FeS , $\text{Al}(\text{OH})_3$, and $\text{Cr}(\text{OH})_3$. This division is known as Group III, and includes Ni^{II} , Co^{II} , Zn^{II} , Mn^{II} , Fe^{II} , Al^{III} , and Cr^{III} . The precipitating reagent is H_2S in a solution alkaline with NH_4OH and containing NH_4Cl . The members of Group III are separated and identified according to procedure for Group III, page 97.

The filtrate from Group III is alkaline, and if to this $(\text{NH}_4)_2\text{CO}_3$ is added, a precipitate of BaCO_3 , SrCO_3 , and CaCO_3 is formed. This division is known as Group IV, and comprises Ba^{II} , Sr^{II} and Ca^{II} . The precipitation reagent is $(\text{NH}_4)_2\text{CO}_3$, in addition to NH_4OH and NH_4Cl previously added. The methods for separating and identifying the members of this group are given in the procedure for Group IV, page 99.

The filtrate from Group IV should not contain any members of the preceding four groups, and may contain Group V in addition to ammonium compounds resulting from compounds formed or added during the analysis.

Group V comprises K^{I} , Na^{I} , and Mg^{II} . The methods for the separation and identification of the members of this group are given in the Group V procedure, page 100.

A test for the NH_4 radical cannot be made in this final solution, but some of the original unknown must be employed for this purpose. The test for the NH_4 radical is given on page 101.

Graphic Method of Representing the Procedure.—In order that the student may become more readily familiar with the procedure, it is advisable that he outline it in some way that will bring out the relationships. One such method is represented below. The same general plan may be employed for the individual groups.

Unknown Solution Add HCl				
Precipitate Group I	Filtrate Groups II, III, IV, V Proper acid conditions + H ₂ S			
	Precipitate Group II	Filtrate Groups III, IV, V Proper conditions, NH ₄ OH, NH ₄ Cl + H ₂ S		
		Precipitate Group III	Filtrate Groups IV, V Proper conditions (NH ₄) ₂ CO ₃	
			Precipitate Group IV	Filtrate Group V

B. Qualitative Procedure for Basic Constituents

GROUP I

Procedure.—Place 10 ml of the solution to be analyzed in a small beaker, add an equal volume of distilled water, and then add 6M HCl drop by drop, stirring constantly, until precipitation seems to be complete. Add 2 ml more of HCl. (If no precipitate forms, the absence of silver, mercury in the mercurous form, and large amounts of lead is indicated. Proceed directly to Procedure, Group II.) Filter. Test for complete precipitation by adding a few drops of HCl to the clear filtrate. If no more precipitate forms, the precipitation of silver, mercurous mercury, and most of the lead is complete. The precipitate is designated P₁, and the filtrate, F₁. Save the filtrate (F₁) for subsequent treatment (Procedure, Group II), since it may contain metals of other groups.

(a) Wash the precipitate on the filter paper twice, carefully, with cold water. Discard the washings.

(b) Pour 30 ml (in three portions) of boiling hot water upon the precipitate (P₁) on the filter paper. The residue left on the paper is designated P₁₁, and the filtrate F₁₁.

(c) 1. To about 5 ml of the clear filtrate (F_{11}), add a few drops of H_2SO_4 . A white precipitate, $PbSO_4$, indicates the presence of **lead**.

2. To another 5 ml portion of the filtrate (F_{11}), add a few drops of K_2CrO_4 . A yellow precipitate, $PbCrO_4$, confirms the presence of **lead**.

(d) Pour upon the residue (P_{11}) on the filter paper, about 10 ml of 5M NH_4OH . Warm the NH_4OH before pouring on the residue. The residue left on the paper is designated P_{12} , and the filtrate F_{12} .

To the clear filtrate (F_{12}) add HNO_3 until the system is acid to litmus. A white precipitate, $AgCl$, indicates the presence of **silver**.

(e) The insoluble black residue (P_{12}) indicates the presence of **mercurous mercury**.

GROUP II

Procedure.—Neutralize the filtrate (F_1) from Group I with NH_4OH until the solution is slightly alkaline to litmus. Stir during the addition of the NH_4OH . Make the solution just acid with HCl , and evaporate until the volume is less than 30 ml. Transfer to an Erlenmeyer flask, and add 6M HCl , employing 1 ml of the acid to each 10 ml of the solution. Heat nearly to boiling, disregarding any precipitate that may have formed, and pass in H_2S for at least three minutes. Dilute with an equal volume of water, and pass in H_2S again, continuing as long as any precipitate seems to form. Filter. Test the filtrate for complete precipitation. The precipitate is designated P_2 and the filtrate F_2 .

(a) Heat the filtrate (F_2), until all the H_2S is expelled. Reserve for Procedure, Group III. The H_2S is all expelled when a piece of filter paper, moistened with $Pb(C_2H_3O_2)_2$ solution, and held just above the heated liquid, no longer blackens or turns brown.

(b) Wash the precipitate (P_2) with a solution of ammonium nitrate (2%) saturated with H_2S . Transfer the precipitate to a small beaker, add 10 ml of sodium sulphide reagent and heat for about 5 minutes. Add 20 ml of water and filter. The precipitate is designated P_{21} and the filtrate F_{21} . The filtrate (F_{21}) may contain compounds of Hg , As , Sb , and Sn^1 and is treated later according to (f) on page 95.

¹ These metals are sometimes designated Group IIB.

(c) The precipitate (P_{21}) may contain PbS , Bi_2S_3 , CuS , and CdS ¹. Wash this precipitate thoroughly with water containing a little H_2S . Transfer the precipitate to an evaporating dish, and cover with 3M HNO_3 , and heat gently. (This is obtained by adding an equal amount of water to your desk reagent.) A little free sulphur may remain floating on top of the solution. Add 2 ml of concentrated H_2SO_4 , and evaporate until white fumes of SO_3 appear. Cool, and pour into a beaker containing about 10 ml of water, washing every bit of the contents of the evaporating dish into the beaker. Allow to stand for one minute before filtering. The precipitate is designated P_{22} and the filtrate F_{22} . Treat the filtrate F_{22} according to (d).

The precipitate (P_{22}) may be $PbSO_4$. Wash with water containing a few drops of H_2SO_4 . Pour over the precipitate on the filter paper about 10 ml of a hot solution of ammonium acetate, catching the filtrate in a test tube. Test this filtrate for **lead** as in (c) of procedure, Group I.

(d) Make the filtrate (F_{22}) just alkaline with NH_4OH . Add 2 ml of the NH_4OH in excess. Filter. The precipitate is designated P_{23} , and the filtrate F_{23} . Wash the precipitate with water containing a small amount of NH_4OH . The filtrate (F_{23}) may contain Cu (indicated by a blue solution) and Cd . The precipitate (P_{23}) may be $Bi(OH)_3$.

Divide the filtrate (F_{23}) into two portions and test as follows:

1st portion.—Acidify with acetic acid and add a few drops of $K_4Fe(CN)_6$. A chocolate colored precipitate confirms the presence of **copper**.

2nd portion.—Add KCN solution until the blue color disappears. Pass in H_2S . A yellow precipitate indicates **cadmium**. (If a black precipitate forms, filter and dissolve the precipitate in hot dilute H_2SO_4 (2 ml of the desk reagent to 8 ml of water). Dilute with 10 times the volume of water, and pass H_2S into the solution. A yellow precipitate indicates cadmium.)

(e) Upon the precipitate (P_{23}) on the filter paper, pour a fresh solution of sodium stannite.² A black deposit indicates **bismuth**.

(f) The filtrate (F_{21}) may contain the complex thiosalts, Na_3AsS_4 , Na_3SbS_4 , Na_2SnS_3 , and Na_2HgS_2 . Add 6M HCl to this

¹ Sometimes designated Group IIA.

² *Preparation of Sodium Stannite Solution.*—Fill the curved portion of your test tube with $SnCl_2$ solution, and add $NaOH$ solution, drop by drop, with frequent stirring, until the precipitate first formed just dissolves. Dilute with an equal volume of water and employ as the test for bismuth.

filtrate until the solution is slightly acid. Filter. Discard the filtrate. The precipitate is designated P_{24} . It may contain S, HgS , As_2S_5 , Sb_2S_5 , and SnS_2 . Dry the precipitate by pressing between two filter papers and removing as much of the water as possible. Peel the dried precipitate off the filter paper, and transfer to a small beaker. Add 10 ml of 12M HCl and heat until no more H_2S is evolved. Add 10 ml of water and filter. The precipitate is designated P_{25} and the filtrate F_{25} . The precipitate may contain HgS and As_2S_5 , and the filtrate may contain $SbCl_3$ and $SnCl_4$.

(g) Treat the precipitate (P_{25}) with 10 ml of 10M NH_4OH . Filter. A black residue indicates mercury, and the filtrate may contain arsenic. The black residue is designated P_{26} and the filtrate F_{26} .

1. Place the black residue (P_{26}) in a small beaker, and add 5 ml of concentrated HCl . Heat and add $KClO_3$ crystals, a few at a time, until the black residue has disappeared. Boil to remove the excess chlorine. Add a few drops of the solution to half a test tube of dilute $SnCl_2$ solution. (Dilute the shelf reagent with three times its volume of water.) A white precipitate which later turns grey or black indicates **mercury**.

2. Acidify the filtrate (F_{26}) containing arsenic with 6M HNO_3 . Boil until the solution is clear. A yellow precipitate of As_2S_3 may appear, and then be oxidized to form arsenic acid. Divide the solution into two portions.

1st portion.—Add to 5 ml of this portion an equal volume of ammonium molybdate solution. Keep the test tube hot by immersing in a beaker of boiling water for five minutes. A yellow precipitate indicates **arsenic**.

2nd portion.—Add to this portion a drop of phenolphthalein solution. Make the solution just alkaline with NH_4OH . Add a few drops of $AgNO_3$ solution. Add carefully, drop by drop, a dilute solution of acetic acid until the addition of one drop causes the color of the phenolphthalein to fade. A brown precipitate of Ag_3AsO_4 indicates the presence of **arsenic**.

(h) The filtrate (F_{25}) may contain compounds of Sb and Sn. Add 30 ml of water. Heat just to boiling, and maintain this temperature by immersing in a beaker of boiling water. Pass in H_2S for a few minutes. An orange precipitate of Sb_2S_3 forms quite soon, which indicates the presence of **antimony**.

Filter off the Sb_2S_3 and reject. The filtrate may contain $SnCl_4$ with traces of antimony. Divide into two portions.

1st portion. Add 8 ml of water, *cool*, and pass H_2S . A bright yellow precipitate of SnS_2 indicates the presence of **tin**. If the amount of tin is small, only a bright yellow coloration may be formed.

2nd portion. To this portion, add 4 iron brads and enough HCl to insure a rapid evolution of hydrogen. Allow to stand for 10 minutes, then filter rapidly into a test tube. Add a few drops of HgCl_2 immediately. A white or greyish white precipitate indicates the presence of **tin**.

GROUP III

Procedure.—To the filtrate (F_2) from Group II (a), page 94, add 2 g of NH_4Cl , and make distinctly alkaline with NH_4OH . Test with litmus. Pass in H_2S until precipitation seems complete. Filter, and test the filtrate for complete precipitation. The precipitate is designated P_3 and the filtrate F_3 . Acidify the filtrate (F_3) with acetic acid, and evaporate to 10 ml. Filter if not clear, and reserve for Procedure, Group IV.

(a) Wash the precipitate (P_3), which may contain CoS , NiS , MnS , ZnS , Fe_2S_3 , FeS , $\text{Al}(\text{OH})_3$, and $\text{Cr}(\text{OH})_3$, with water containing a little NH_4OH and H_2S . Transfer the precipitate to a 100 ml beaker and add 25 ml of M HCl . Filter, and reserve the filtrate. The precipitate is designated P_{31} , and the filtrate F_{31} .

(b) The precipitate (P_{31}) may contain CoS and NiS . Wash with cold water. Test the precipitate for cobalt and nickel by a borax bead heated in the oxidizing flame. Transfer the precipitate to an evaporating dish, and add 5 ml of 6M HCl . Boil and add 6M HNO_3 , drop by drop, until the black precipitate disappears. If a residue of sulphur remains, remove it with a stirring rod. Neutralize the solution with NaOH . Acidify with acetic acid, and add 3 ml of acetic acid in excess. Divide into two portions.

1st portion.—Test for nickel by adding a few drops of a solution of dimethylglyoxime. Make alkaline with NH_4OH and boil. A red precipitate indicates **nickel**.

2nd portion.—To about 3 ml of water in a test tube, add an equal volume of solid potassium nitrite. Saturate with KCl the solution to be tested for cobalt, by adding a few crystals at a time, and shaking until no more will dissolve. Into the solution of potassium nitrite, pour an equal volume of the solution to be

tested. Let stand for 30 minutes, if a precipitate does not appear at once. A yellow precipitate indicates the presence of **cobalt**.

(c) The filtrate (F_{31}) may contain $FeCl_2$, $MnCl_2$, $AlCl_3$, $CrCl_3$, $ZnCl_2$, and an excess of H_2S and HCl . Boil to remove all of the H_2S . Add 1 ml of 6M HNO_3 , and boil for a couple of minutes to oxidize all the iron. Add NH_4OH until the solution is alkaline. Add 10 ml of $(NH_4)_2CO_3$ and heat to boiling. Filter. The precipitate (P_{32}) may contain $Fe(OH)_3$, $MnCO_3$, $Al(OH)_3$, and $Cr(OH)_3$. The filtrate (F_{32}) may contain complex compounds of zinc, and traces of nickel and cobalt.

(d) Treat the filtrate (F_{32}) with acetic acid in excess. Pass in H_2S . A white precipitate indicates **zinc**. A black precipitate may be due to traces of nickel and cobalt.

(e) Place the precipitate (P_{32}) in a small beaker and dissolve in 3M HCl . The solution may contain the chlorides of iron, manganese, aluminum, and chromium. Evaporate to about 1 ml and dilute with 10 ml of water. Neutralize with $NaOH$ and add 5 ml in excess. Cool. Add about 2 grams of Na_2O_2 , slowly and with constant stirring, using a glass rod. Boil for one minute, continuing to stir constantly. Dilute with 10 ml of water, and filter. The precipitate (P_{33}) may contain $MnO(OH)_2$ and $Fe(OH)_3$. The filtrate (F_{33}) may contain Na_3AlO_3 and Na_2CrO_4 .

(f) The precipitate (P_{33}) is placed in a small beaker. Pour upon it 10 ml of 6M HNO_3 . Boil. Add solid $KClO_3$, a little at a time, until no more precipitate forms. Dilute with an equal volume of water. Filter. The precipitate may be $MnO(OH)_2$ together with MnO_2 , and is designated P_{34} . The filtrate is designated F_{34} . The precipitate (P_{34}) is tested for manganese by making a sodium carbonate bead, and dipping the latter first into the precipitate, and then into powdered $KClO_3$. The bead is then fused again. A light green coloration of the bead indicates **manganese**.

The filtrate (F_{34}) may contain $Fe(NO_3)_3$. Add NH_4OH in excess. A brick red precipitate of $Fe(OH)_3$ indicates **iron**. Filter off the $Fe(OH)_3$ and dissolve in a small amount of dilute HCl . Add an equal volume of water, and divide into two parts. Add a few drops of a solution of $KCNS$ to one part. A deep red coloration indicates **iron**. To the other part, add 1 ml of $K_4Fe(CN)_6$ solution. A deep blue precipitate indicates **iron**.

(g) The filtrate (F_{33}) is tested for Al and Cr as follows: Acidify with HNO_3 and neutralize with NH_4OH . Boil for a minute.

Filter. A white gelatinous precipitate (P_{35}) indicates **aluminum**. The filtrate F_{35} , if yellow, may contain chromium.

1. If the precipitate P_{35} is small, it may be silicic acid which has been introduced as an impurity in the various alkaline reagents used. In such cases, pour a few ml of concentrated HCl through the filter paper, and evaporate the filtrate **just to dryness**, in a small beaker. **Cool**, and add to the residue, 5 ml of water and a few drops of dilute HCl. **Warm**, and filter. To the filtrate add enough alizarin solution to produce a light straw color. Add an excess of NH_4OH , and heat to boiling. The formation of a precipitate which absorbs the red dye, leaving a comparatively colorless filtrate, indicates **aluminum**.

The following method may be used instead of that just given for the identification of aluminum. Dissolve the white gelatinous precipitate in 5 ml of M HCl. Add 5 ml of 3M $NH_4C_2H_3O_2$ and 5 ml of 0.1% solution of the ammonium salt of aurin tricarboxylic acid. Mix thoroughly to allow a lake to form. Add NH_4OH containing a little $(NH_4)_2CO_3$ until the system is alkaline. A bright red lake in the final alkaline system indicates the presence of aluminum.

2. Divide the filtrate F_{35} into two portions.

1st portion.—Acidify with acetic acid, and add 2 ml in excess. Add a few drops of lead acetate solution. A yellow precipitate of $PbCrO_4$ confirms the presence of **chromium**.

2nd portion.—Cool. Add 1 ml of ether, 1 ml of H_2O_2 , acidify with H_2SO_4 , and shake. A blue color, owing to the presence of perchromic acid in the ether layer, proves the presence of **chromium**.

GROUP IV

Procedure.—Make the filtrate (F_3) from Group III alkaline with NH_4OH , and add 5 ml of $(NH_4)_2CO_3$. Heat until the precipitate becomes crystalline. Test for complete precipitation. Filter. Wash the precipitate with a dilute solution of $(NH_4)_2CO_3$. The precipitate is designated P_4 , and the filtrate F_4 . Add 1 ml of $(NH_4)_2SO_4$ and 1 ml of $(NH_4)_2C_2O_4$ solutions to the filtrate (F_4), and reserve for Procedure, Group V.

(a) The precipitate (P_4) produced by ammonium carbonate may be $BaCO_3$, $CaCO_3$, and $SrCO_3$. Make preliminary flame and spectroscopic tests. Pour hot 5M acetic acid upon the precipitate, in 2 ml portions at a time, until the precipitate is all dissolved.

Dilute to about 50 ml. Add K_2CrO_4 solution, drop by drop, until no more precipitate seems to form. Add a few drops in excess. Boil, and filter while hot. Wash the precipitate with hot water. The filtrate is designated F_{41} and the precipitate P_{41} . The precipitate may be $BaCrO_4$, indicating the presence of barium.

Dissolve the precipitate (P_{41}) in 3M HCl. Make confirmatory flame and spectroscopic tests for Ba.

(b) The filtrate (F_{41}) may contain salts of calcium and strontium, together with an excess of K_2CrO_4 . Make alkaline with NH_4OH , and add $(NH_4)_2CO_3$ until no more precipitate forms. Warm and filter, and wash the precipitate with small amounts of hot water until the washings are no longer yellow. Discard the washings. The precipitate (P_{42}) may consist of $SrCO_3$ and $CaCO_3$.

(c) Dissolve the precipitate (P_{42}) in a little warm acetic acid. Dilute the solution to about 10 ml, and divide it into two portions.

1st portion.—To 5 ml of a saturated solution of $CaSO_4$ add 1 ml of this solution. A clouding or white precipitate indicates **strontium**.

2nd portion.—If Sr is present, add to this portion 5 ml of $(NH_4)_2SO_4$ solution. Warm. Test for complete precipitation, and filter. Wash with water. The precipitate (P_{43}) may be $SrSO_4$, indicating the presence of **strontium**, and the filtrate (F_{43}) may contain Ca.

To confirm the presence of Sr in P_{43} , digest the precipitate with Na_2CO_3 solution. Filter and wash. Dissolve the precipitate in a small amount of HCl and examine with the spectroscope. (See table in appendix for color of flame.) Make the filtrate (F_{43}) alkaline with NH_4OH , and add $(NH_4)_2C_2O_4$ solution. Warm, and allow to settle. A white crystalline precipitate indicates the presence of **calcium**. Filter, dissolve in a little HCl, and confirm by flame color and the spectroscope.

GROUP V

Procedure.—The filtrate from Group IV may contain the salts of Na, K, and Mg. An excess of ammonium salts is usually present. If a small amount of precipitate has formed while the filtrate (F_4) has been standing, filter, and reject the precipitate. To the clear, cold filtrate, add 5 ml of secondary ammonium orthophosphate $(NH_4)_2HPO_4$. Shake or stir vigorously, and add 5 ml of 10M NH_4OH . If a precipitate does not appear at once, let the solution stand for 30 minutes. The precipitate is $MgNH_4PO_4$, indicating

the presence of **magnesium**. Filter. The filtrate (F_5) may contain salts of Na and K.

Evaporate the filtrate (F_5) to dryness in an evaporating dish, by adding small portions at a time. (Do not add liquid to a hot, dry dish.) When all the filtrate has been evaporated, heat the dry residue in the dish more and more strongly, until all fumes of ammonium salts cease. **This must be done under the hood.** The residue in the dish may be salts of Na and K. Examine this residue by the spectroscope and by the flame reaction for potassium. Add 4 ml of water, and get all the residue into solution. Filter and reject any residue on the filter paper. Divide filtrate into two portions.

1st portion.—Add 5 ml of perchloric acid solution, and evaporate until dense white fumes of perchloric acid arise from the dish. *Cool thoroughly.* Add 10 ml of 95 per cent alcohol and stir. A white crystalline precipitate indicates *potassium*. Filter. Dissolve in hot water and check with the spectroscope.

2nd portion.—Make just alkaline with KOH. Add an equal volume of alkaline potassium pyroantimonate solution. Let stand for at least half an hour. A white crystalline precipitate indicates **sodium**.

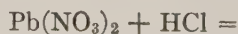
Alternate Test for Potassium.—1st portion may be treated with 1 ml of sodium cobaltinitrite solution, and allowed to stand. A yellow crystalline precipitate indicates the presence of **potassium**.

Test for Ammonium Salts.—Place about 5 ml of the original unknown solution in a 50 ml beaker. Add about the same amount of concentrated NaOH solution. Cover the beaker with a cover glass, on the underside of which must be pasted a piece of moist red litmus paper. Heat the solution. If ammonium salts are present, the NH_3 evolved will turn the red litmus blue, thus indicating the presence of **ammonium** salts in the original unknown.

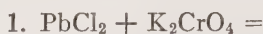
C. Equations Indicating the Reactions Occurring in the Procedure

GROUP I—REACTIONS

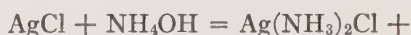
The metals of Group I are precipitated as chlorides by HCl. (We can assume the metals to be in solution as nitrates.)



The PbCl_2 is dissolved in hot water, and tested for the presence of Pb.



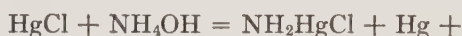
The Ag and the Hg remain on the filter paper as chlorides, and are separated by means of NH_4OH , which reacts with AgCl as follows:



This filtrate is treated with HNO_3 , which reprecipitates the AgCl .



The HgCl remaining on the filter paper is turned black, owing to the formation of a mercury complex and black metallic mercury.

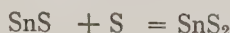


GROUP II—REACTIONS

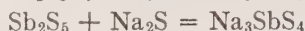
The metals of Group II are left in the filtrate from the Group I precipitation. The metals of Group II are precipitated as sulphides in an acid solution.



Separation of the Subgroups.—The precipitated sulphides are filtered off, and the HgS , As_2S_3 , Sb_2S_3 , and SnS are dissolved in the sodium sulphide reagent. The filtrate contains the complex thiosalts of Hg, As, Sb, and Sn. (The Na_2S - NaOH reagent contains Na_2S together with some S and NaOH .) The Sn, As, and Sb are first oxidized by the sulphur.

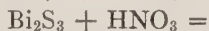
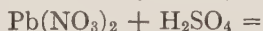


These sulphides, together with the sulphide of Hg, are then dissolved by the Na_2S - NaOH reagent to form the thiosalts. (See text pages 361 and 364.)

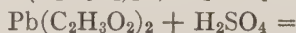
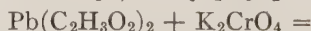


The solution is filtered and the thiosalts of As, Sb, Sn and Hg are separated from the sulphides of Pb, Bi, Cu, and Cd.

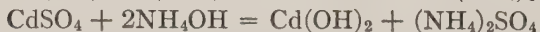
1. The precipitate containing the sulphides of Pb, Cu, Cd, and Bi is treated with HNO_3 and then with H_2SO_4 which converts them to the sulphates. PbSO_4 is insoluble, while the others are soluble in water, and are filtered off.



The PbSO_4 is dissolved in $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$, and tested for Pb.

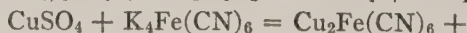
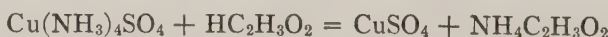


2. The filtrate from 1 is then made alkaline with NH_4OH . The Cu and Cd are dissolved as ammonia complexes of CuSO_4 and CdSO_4 , while the Bi is precipitated as $\text{Bi}(\text{OH})_3$.

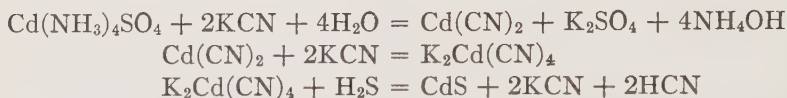


The filtrate (a) is divided into two portions.

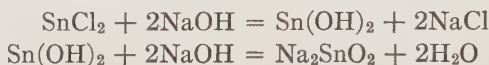
1st portion.—The solution is acidified with acetic acid, and tested for copper with $\text{K}_4\text{Fe}(\text{CN})_6$.



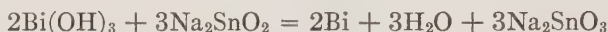
2nd portion.—KCN solution is then added until the color of the blue copper ammonia complex is destroyed. The solution is then treated with H_2S , which precipitates yellow CdS if Cd is present.



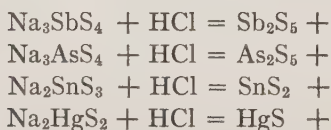
The precipitate of $\text{Bi}(\text{OH})_3$ is treated with sodium stannite the solution which is prepared as follows:



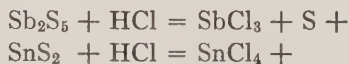
The sodium stannite solution reduces the $\text{Bi}(\text{OH})_3$ to metallic bismuth, which colors the filter paper black.



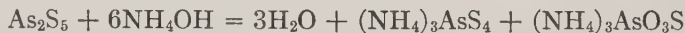
3. The filtrate from separation of the subgroups may contain thiosalts of As , Sb , Sn and Hg . This solution is acidified with HCl , thus precipitating the sulphides of these metals.



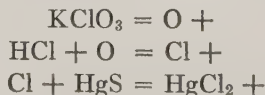
This precipitate is treated with HCl (Sp. Gr. 1.2) which dissolves the Sb and Sn .



(a) The residue from the above treatment contains Hg , As , as sulphides. This is treated with NH_4OH , which dissolves the As , leaving the Hg as HgS .

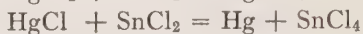


(1) The HgS is dissolved in a concentrated solution of HCl , to which some KClO_3 is added.



This solution of HgCl_2 is divided into two portions.

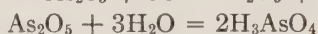
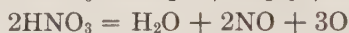
1st portion.— SnCl_2 is added, which precipitates white HgCl and black Hg .



2nd portion.—Add a piece of bright Cu foil. Hg is deposited on the foil.



(2) The arsenic filtrate is acidified. This precipitates As_2S_5 , which dissolves to form arsenic acid.

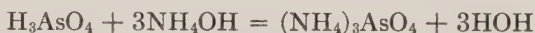


The solution containing the arsenic acid is divided into two portions.

1st portion.—Ammonium molybdate solution is added which precipitates yellow ammonium arsenomolybdate, if As is present.



2nd portion.—The solution is just neutralized and AgNO_3 added, which precipitates brown Ag_3AsO_4 .



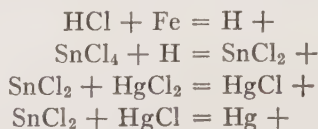
(b) The Sb_2S_3 is separated from the SnS_2 by the difference in solubility, in a solution of HCl . The precipitate of Sb_2S_3 indicates the presence of Sb . The Sn passes into the filtrate as SnCl_4 . This is divided into two portions.

1st portion.— H_2S is passed into the cold solution of SnCl_4 which precipitates yellow SnS_2 .



2nd portion.—The SnCl_4 is reduced by nascent hydrogen ($\text{HCl} + \text{Fe}$) to SnCl_2 which is in turn tested for Sn by the addition

of HgCl_2 solution. A grey or black precipitate of HgCl and Hg indicates the presence of Sn .

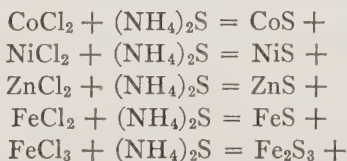


GROUP III—REACTIONS

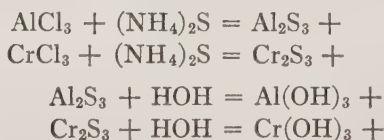
The metals of Group III are precipitated by $(\text{NH}_4)_2\text{S}$, which is prepared in solution as follows:



The following equations represent the precipitation of Group III.



Aluminum and chromium sulphides hydrolyze readily.



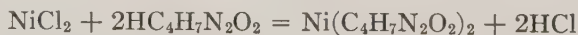
1. Zn , Mn , Fe , Al , and Cr are separated from Ni and Co by the difference in the solubilities of NiS and CoS in M HCl . Aqua regia is prepared as follows (See text, page 284):



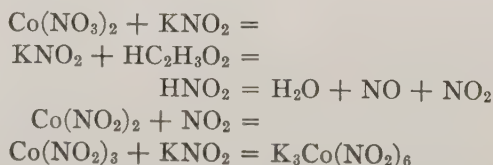
NiS and CoS are dissolved in this aqua regia.



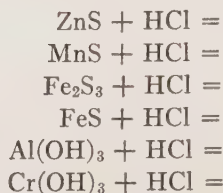
1st portion is tested for Ni as follows:



2nd portion is tested for Co as follows:

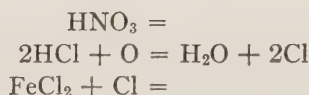


The sulphides of Zn, Mn, Fe, Al, and Cr which were precipitated in the preliminary precipitation are dissolved in HCl as follows:

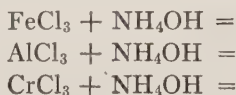


These soluble chlorides were filtered off from the insoluble sulphides of Ni and Co.

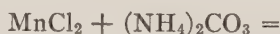
2. HNO_3 is again added, to oxidize the Fe to FeCl_3 , as follows:



The hydroxides of Fe, Al, and Cr are precipitated by the addition of NH_4OH .

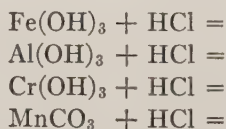


Likewise, the carbonate of Mn is precipitated by the addition of $(\text{NH}_4)_2\text{CO}_3$.

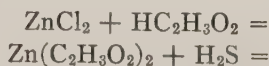


The Zn, which existed in solution as the chloride, passed into the filtrate as an ammonia complex of ZnCl_2 , and is separated from the precipitated Fe, Al, Cr and Mn.

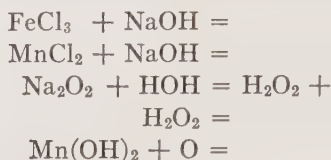
3. The precipitated Fe, Al, Cr and Mn is then dissolved in HCl.



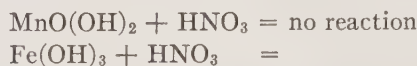
4. The ZnCl_2 filtrate is treated with acetic acid, and tested for Zn by precipitation of the sulphide.



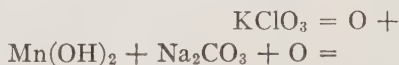
5. The Fe and Mn are precipitated with NaOH and Na₂O₂.



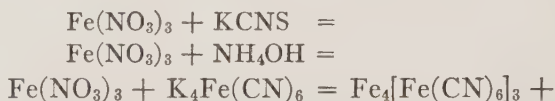
This precipitate containing Fe and Mn is treated with HNO₃.



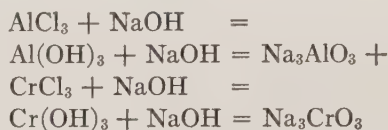
6. Test for Mn is made by the Na₂CO₃ bead. KClO₃ is added to oxidize the Mn.



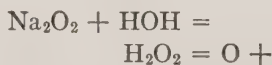
7. The tests for Fe are made with NH₄OH, KCNS, and K₄Fe(CN)₆



8. The Al and Cr are dissolved from the precipitated Fe and Mn, in the treatment with NaOH and Na₂O₂.



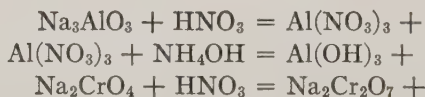
Oxygen is furnished by the Na₂O₂.



This oxidizes the chromite to the chromate.

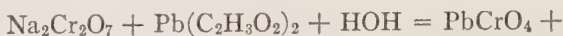


The Al and Cr in the filtrate are treated with HNO₃.



9. The precipitate of Al(OH)₃ is then confirmed by the absorption of the organic dye, alizarin.

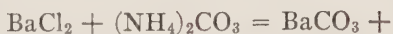
10. The filtrate containing the Cr is tested with $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$, and with H_2O_2 in acid solution.



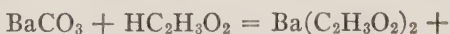
$\text{Na}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{O}_2 =$ blue perchromic acid which decomposes in aqueous solution but is stable in ether solution (no equation required).

GROUP IV—REACTIONS

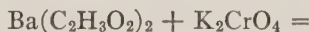
The metals of Group IV are precipitated as carbonates by the addition of $(\text{NH}_4)_2\text{CO}_3$.



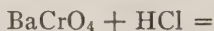
1. This precipitate contains Ba, Sr, and Ca as carbonates, which are insoluble and are filtered off. Preliminary flame and spectroscopic tests are made (refer to notes on spectroscopic and flame tests) and the remaining precipitate is dissolved in acetic acid.



The barium is separated from the Ca and the Sr by precipitation as the chromate.



The precipitate of barium chromate is dissolved in HCl.



The solution of BaCl_2 is tested by flame and spectroscopic tests to confirm the presence of barium.

2. The filtrate containing an excess of K_2CrO_4 is made alkaline, and the Ca and Sr reprecipitated as carbonates in order to remove the excess of K_2CrO_4 .

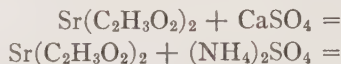


The filtrate contains the excess of K_2CrO_4 which is rejected.

3. The precipitate consisting of the carbonates of Ca and Sr is dissolved in acetic acid.



The Sr is precipitated from this solution as the sulphate, by the addition of CaSO_4 and $(\text{NH}_4)_2\text{SO}_4$.



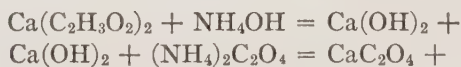
4. The precipitate contains the Sr as the sulphate. It is converted to the carbonate by digestion with Na_2CO_3 .



The precipitate is then dissolved in HCl , and the solution of the chloride used for confirmatory flame and spectroscopic tests.



5. The calcium is precipitated as the oxalate by the addition of $(\text{NH}_4)_2\text{C}_2\text{O}_4$



GROUP V—REACTIONS

1. Magnesium is precipitated by means of secondary ammonium ortho phosphate.

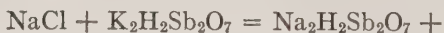


2. The filtrate contains Na and K which are identified by their flame and spectroscopic tests.

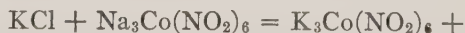
The reaction with HClO_4 is



The reaction with alkaline potassium pyroantimonate is



The reaction with sodium cobaltinitrite solution is



Test for Ammonium Salts.—In the original unknown solution, we can assume the salt to be in the form of a nitrate.



II. ACIDIC CONSTITUENTS

The tests for the metals should be practically completed before the analysis for the acidic constituents is begun, since a knowledge of the metals present gives considerable information concerning the possible acid radicals. Of course, as the metals are determined

group by group, the tests for the acid radicals may be begun soon after the first groups have been worked through. It is also true that a knowledge of the acid radicals assists in checking up the metals that can or cannot be present. For instance, if the halides are shown to be present, this excludes the presence of Group I. Conversely, if Group I is found to be present, this excludes the presence of chlorides, bromides, and iodides.

A. Preliminary Examination of Solid Unknown Systems

A few preliminary tests of solid substances for acid radicals are desirable, and should be carried out before beginning the systematic tests for the acidic constituents.

The following special tests should be made on the solid unknown:

(a) Examine carefully with the unaided eye. Use a microscope if available. Note the form, color, luster, and any peculiarity in appearance of the system. Grind to a fine powder, if possible, since it is more soluble in this form.

(b) Heat a small quantity of the system in a hard glass test tube. Note carefully all the changes that occur, such as:

1. Decrepitation. If decrepitation takes place, what substance may be present?

2. Gases given off. Colored or colorless? Odor? Odorless? What do the gases signify?

3. Sublimation. What does this show?

4. Water given off? What does this indicate?

5. Marked change in appearance. Due to what?

(c) Treat a small amount of the solid with dilute HCl. What evidence is there of a chemical reaction?

1. Does it effervesce? Test gas with lime water. Cloudy drop indicates CO_2 .

2. Odor of gas? SO_2 indicates a sulphite. H_2S indicates sulphides.

3. Color of gas? Brown indicates **nitrites**.

4. By means of a platinum wire, hold some of the solution in a colorless flame. Note colors imparted to the flame. (See table in Appendix.) Verify by the spectroscope.

(d) Apply the Borax Bead Test. Note color of bead when hot and cold. Indicates presence of what? (See table in Appendix for coloration of beads.)

(e) Test for ammonium compounds. Place a small amount of the unknown solid system in a beaker. Add 5 ml of water and mix thoroughly. Add 5 ml of a NaOH solution. Cover with a cover glass, on the underside of which must be pasted a piece of red litmus paper. Heat. If the litmus changes color, the presence of **ammonium compounds** in the original sample is indicated.

(f) Treat with concentrated H_2SO_4 . To a small amount of the solid unknown, in a test tube, cover with concentrated H_2SO_4 and heat. Note carefully any changes.

1. Test gases with moist blue litmus paper. If it turns red, halogen acids or acid anhydrides are present.

2. Test for CO_2 with a drop of lime water, suspended by a glass rod or platinum wire. A clouding of the drop indicates a **carbonate**.

3. Test for H_2S by holding a piece of filter paper, moistened with lead acetate, in the escaping gas. A black or brown coloration indicates a **sulphide** in the original system.

4. If the gas rising in the test tube is brown, it may be (1) bromine, indicating a **bromide**, (2) nitrogen dioxide (NO_2), indicating a **nitrate** or **nitrite**, (3) chromyl chloride (CrO_2Cl_2) indicating a **chloride** and a **chromate** in the original system.

5. If the gas rising is purple, an **iodide** is indicated in the original system.

6. Test for a borate by pouring the cold contents of the test tube into an evaporating dish. Cover with alcohol, stir, ignite the alcohol by means of the alcohol on a stirring rod, which is ignited in the flame and used as a lighter. Extinguish the burning alcohol, and relight. Repeat several times. A green tinge to the alcohol flame indicates a **borate** present in the original system.

7. If, upon adding H_2SO_4 and heating, there is a crackling sound evidencing small explosions, the presence of *chlorates* in the original system is indicated.

B. Systematic Examination of Unknown System for Acidic Constituents

The acidic constituents may be conveniently classified into three general divisions, based upon the treatment with AgNO_3 and $\text{Ba}(\text{NO}_3)_2$, but before making the tests for the acidic constituents, it is advisable to remove all the basic or metallic constituents.

A neutral solution of the unknown is preferred, but an acid solution must often be employed. The solution of the unknown is neutralized and boiled with a concentrated Na_2CO_3 solution until all ammonia is given off, if any appears. Filter. The filtrate will contain the sodium salts of the acid constituents, and an excess of the sodium carbonate. This is designated the *prepared solution* and is to be used in the tests for the acidic constituents. Divide the solution into two portions and save one portion for special tests. Acidify the other portion of the solution with HNO_3 , and boil to expel the CO_2 . Make alkaline with NH_4OH , and boil off the excess. Filter, if necessary. Divide this into two portions.

1st portion.—Acidify with HNO_3 a portion of the prepared solution, and add AgNO_3 . A precipitate indicates the acidic constituents of Division I, which comprises those whose silver salts are insoluble in dilute HNO_3 , and includes **chlorides, bromides, iodides, cyanides, and sulphides**.

2nd portion.—To the other portion of the prepared solution, add $\text{Ba}(\text{NO}_3)_2$, which, from neutral solutions, precipitates the acidic constituents of Division II, which comprise:

a. Those whose barium salts are insoluble in HCl solutions. These are **sulphates, fluorides, and fluosilicates**.

b. Those whose barium salts are insoluble in water, that is, are precipitated from neutral solutions. These are **sulphites, carbonates, oxalates, phosphates, thiosulphates, chromates, borates, arsenates, arsenites**.

There is an additional division designated Division III, which comprises those acidic constituents whose silver and barium salts are soluble in water. These include the **nitrates, chlorates, nitrites and acetates**.

C. Special Tests for Acidic Constituents

The detection of the acidic constituents is largely an individual matter, and they are not determined, as are the basic constituents, by a definite procedure of separation, but are tested for by individual specific tests. Many of these have been indicated above, under the preliminary examination of an unknown solid system. These may be restated under the grouping that has just been given. It is best to use the prepared solution for these tests, but the water and the acid solutions of the unknown can sometimes be employed.

Division I. Chlorides, Bromides and Iodides.—These are precipitated from an acid solution by the addition of an excess of AgNO_3 .

(a) A white precipitate indicates a **chloride** or **bromide**, and a yellow precipitate an **iodide**. Test the solubility in ammonium hydroxide and by the action of light. (Exercises 35 and 36.)

(b) To distinguish these, 2 ml of CCl_4 are added to 5 ml of the unknown acidified solution, and then chlorine water added, a little at a time, and the system shaken thoroughly. (See Exercise 51.)

1. A pink coloration indicates the presence of an **iodide**.

2. A brown coloration indicates the presence of a **bromide**.

3. Since iodine is liberated first by the addition of chlorine water, an excess of chlorine water will oxidize the iodine to iodic acid, which is colorless, and then the bromine will be liberated, if present. So in this test, if the pink color appears, indicating iodine, add chlorine water until the system becomes colorless, and then a few drops in excess, when bromine will be liberated, coloring the CCl_4 brown, if present.

Division II.—The special tests for the most common acidic constituents of this group are:

(a) **Sulphates.**—To the acidified solution, add $\text{Ba}(\text{NO}_3)_2$, drop by drop, until no further precipitation occurs. Test the solubility of the precipitate in HCl or in HNO_3 . An insoluble precipitate indicates a **sulphate**. (See Exercise 54.)

(b) The precipitate formed when $\text{Ba}(\text{NO}_3)_2$ is added to the neutral water solution consists of barium salts insoluble in neutral water solutions, but soluble in dilute acid solutions. These include carbonates, sulphites, phosphates, thiosulphates and borates.

1. **Carbonates.**—To the unknown solution or solid, add dilute HCl or H_2SO_4 . An effervescence indicates the presence of a carbonate, and the gas given off is CO_2 . To prove this, hold a drop of lime water on a stirring rod, or a platinum wire, in the issuing gas. A clouding of the drop indicates CO_2 .

2. **Sulphites.**—(See Exercise 53.) Acidify the solution with H_2SO_4 , heat, and test the escaping gas with a drop of KMnO_4 in the loop of a platinum wire.

3. **Thiosulphates.**—The addition of dilute HCl to a solution of a thiosulphate results in the liberation of SO_2 and the precipitation of sulphur.

4. *Borates*.—Evaporate the solution to be tested, which must be free from copper and barium, to dryness. Add about 2 ml of concentrated H_2SO_4 . To the cool solution, add 5 ml of alcohol and stir thoroughly. By means of the stirring rod, ignite the alcohol. Notice the color of the flame. If it is green, this is due to ethyl borate $(\text{C}_2\text{H}_5)_3\text{BO}_3$, which confirms the presence of a borate. Extinguish the flame, and relight it. Repeat several times to be sure of your results.

5. *Phosphates*.—Acidify the solution of the unknown with HNO_3 , heat, and add about an equal volume of the ammonium molybdate solution. Place in a beaker of warm water, and allow to stand for half an hour. A yellow precipitate indicates the presence of a **phosphate**. (See Exercise 65.)

Division III.—Of the acidic constituents of this division, the **nitrate radical** is the only one of special interest, and the test for this is as follows:

To 5 ml of the aqueous solution of the unknown system, add an equal volume of concentrated H_2SO_4 . Mix, and cool thoroughly. Tip the test tube, and pour very carefully upon this sulphuric acid solution about 5 ml of a solution of ferrous sulphate, so as to not mix them but so that they form two distinct layers. Place the test tube in the rack, and allow to stand for several minutes. The formation of a dark ring between the two layers formed by the two solutions indicates the presence of a **nitrate** in the original system. (See Exercise 62(d).)

III. PREPARATION OF THE SYSTEM FOR ANALYSIS

In the preliminary work, the systems given for analysis are solutions. It frequently happens that the unknown systems to be analyzed are solid. In this case, it is necessary to get them into solution. Often, this is not an easy matter.

It will be recalled that the identification of substances depends upon converting the metallic or basic part, or the nonmetallic or acidic part, of the compound into new compounds which are themselves but slightly soluble in the medium employed. In the case of the metals of Group I, they are converted into chlorides which are but slightly soluble (insoluble) in concentrated HCl solution. Likewise the sulphates of Pb, Ba and Sr are insoluble. This fact is made use of in testing for the SO_4 radical.

When an unknown system is to be analyzed, it is first gotten into solution by dissolving the substances themselves or converting them into soluble compounds. This is not always easy, since many substances are insoluble, while in other cases, they may dissolve, but react as soon as they are in solution, and produce an insoluble substance that precipitates and leaves an insoluble residue. For instance AgNO_3 and BaCl_2 are both soluble in water. When their solutions are mixed, AgCl precipitates. So, if a solid consisting of these two substances is digested with water, there would remain an insoluble residue of AgCl . We might conclude that the original solid was insoluble, since a residue is left after the treatment with water.

A. Insoluble Substances

Insoluble substances are usually considered to be those substances which are insoluble in water, but they also include those substances which are insoluble in acid and alkaline solutions. We have already met these types, and have employed them for the separation and identification of the more common metals and nonmetals with which we have worked in Qualitative Analysis. It would be well to list these, and review the methods of obtaining them as precipitates, and the confirmatory tests employed to identify them. This information is valuable, and aids in identifying the insoluble residues left after the usual means for getting them into solution have failed. For example, PbSO_4 is insoluble in acids; so, if after treating a solid with acids, a residue remains, we may suspect that it might be PbSO_4 . If we remember that PbSO_4 dissolves when treated with acetic acid and ammonium acetate, we can treat the residue, and if it responds to this treatment, and dissolves, we may infer it is PbSO_4 . This can be proved by the usual tests for lead.

The more common insoluble substances comprise: (1) the halides of Pb, Hg', and Ag; (2) the sulphides of the metals below hydrogen in the electrochemical series, and Pb and Sn; (3) the carbonates of the metals, except the alkalis; (4) the sulphates of Pb, Ba, and Sr; (5) the chromates of Pb and Ba; (6) most oxides; (7) metals and alloys; (8) a few less common substances, such as the silicates, stannic acid, and the primary phosphates of the metals, excepting the alkaline metals.

B. Preparation of the Solution for Analysis

Procedure.—The procedure for obtaining a solution of an unknown system which may contain some of these difficultly soluble substances is as follows:

(a) Digest about 2 ml (2 g) of the sample with 30 ml of hot water, using 10 ml portions. Decant the supernatant liquid through a filter paper each time. This removes the substances soluble in water. Mark this solution S_1 , water soluble portion, and save. The residue is designated R_1 .

(b) Treat the residue, R_1 , with 5 ml of dilute 6M HNO_3 , warm, and add more HNO_3 to replace that used up. If there is no apparent action, add a few ml of concentrated HNO_3 . Allow the residue to settle, and decant the solution. Repeat the treatment again. Decant, and place the filtrates together and mark them S_2 , nitric acid solution. The residue is designated R_2 .

(c) Wash the residue, R_2 , and decant the liquid into S_2 (or it may be discarded). To the residue, add about 5 ml 6M HCl and boil for 5 or 10 minutes. If the residue does not disappear, allow to settle and decant. Filter and wash. Save the filtrate, and mark it S_3 , HCl solution. The residue is R_3 .

(d) Digest the residue R_3 with 5 ml of aqua regia. Boil, dilute and filter. Mark the filtrate S_4 , aqua regia solution. The residue is R_4 .

(e) If there is still a residue, R_4 , dry it, and mix it with about 5 times as much fusion mixture (a mixture of equal amounts of Na_2CO_3 and K_2CO_3) designated NaKCO_3 , and a small amount of KNO_3 . Heat this mixture in a covered porcelain crucible until the system is entirely fused, and homogeneous in appearance. Allow the crucible and contents to cool below the boiling temperature of water, and then place them in a beaker. Cover with hot water, boil and stir until the undissolved residue is finely divided. Filter, and wash with hot water. Label this S_5 and test a portion for the sulphate radical according to directions in Exercise 55(a). Cover the residue (R_5) with about 10 ml 6M HCl , or enough to make the solution strongly acid. Heat, stir, filter and wash with hot water. Evaporate to about 5 ml, dilute with 20 ml of water and reserve as S_6 , and label HCl Solution of Fused Mass. Any residue, R_6 , may be tested by special methods for other constituents.

(f) The residue, R_3 or R_4 , may be treated, however, by special methods, instead of proceeding according to the directions in (e).

1. Test a portion of the residues, R_3 or R_4 , for halides of Group I by treating it with hot ammonium hydroxide, and follow the method given according to the regular procedure, page 93.

2. Test a portion of the residues, R_3 or R_4 , for $PbSO_4$ according to the procedure with acetic acid and ammonium acetate, page 116.

3. Test a portion of the residues, R_3 or R_4 , for $BaSO_4$ by boiling them for about 20 minutes in a concentrated solution of Na_2CO_3 . Most of these sulphates will be converted to the carbonates. Add about 10 ml of water, filter, and wash the residue thoroughly. The filtrate may be tested for the SO_4 radical. The residue, which may be $BaCO_3$ and $SrCO_3$, is treated with acetic acid, and tested according to the procedure for Group IV, page 99.

(g) The unknown may now be dissolved in these six different solutions, labeled $S_1, S_2, S_3, S_4, S_5, S_6$. The next procedure is that of seeing whether these can be mixed together without the formation of precipitates, and thus producing one instead of six unknown solutions to analyze. Small portions of these solutions may be first evaporated on a watch glass to ascertain if anything had been extracted by the reagents used. If there is no residue on evaporation, nothing had been extracted, and this particular solution can be discarded.

1. In a test tube, place about 1 ml of S_1 , the water solution, and to this, add 1 ml of S_2 , the nitric acid solution. If they form a homogeneous solution, the two solutions can be added together, forming one unknown solution.

2. In like manner try S_1 and S_3 ; S_2 and S_3 ; S_4 with all three of the others. If these can be mixed without forming a heterogeneous system, they can be all mixed together to form one unknown. But if it is found that these different solutions do not form homogeneous systems when mixed, they will have to be run as separate unknowns.

3. However, before mixing the main bulk of these solutions, it is necessary to evaporate the nitric acid and aqua regia solutions practically to dryness, to get rid of the excess HNO_3 , and take up with water. Concentrated HNO_3 decomposes H_2S , with the precipitation of sulphur in a fine state of division which may run through the filter paper and interfere with the analysis.

APPENDIX

FLAME COLORATIONS AND BEAD TESTS
METHODS OF PREPARATION OF REAGENTS

FLAME COLORATIONS AND BEAD TESTS

1. Flame Colorations.—Certain metals, Na, K, Li, Ba, Sr, and Ca, give characteristic flame reactions. When a flame test of these metals is desired, they are converted to the chlorides by treating their solid salts with concentrated HCl, since the chlorides are among the most volatile salts of the metals.

A platinum wire is provided with a loop at the end. This is dipped into the solution, and then introduced into the clear, oxidizing part of the flame. The flame is then observed with the unaided eye. In the case of potassium, it is well to observe the flame through several thicknesses of cobalt glass, which absorbs the sodium light. The characteristic colors of the flames are given in the following table.

It is better however, to employ a spectroscope to view the flame, as distinct characteristic lines in the spectrum indicate the metal present.

2. Borax Beads.—Borax beads are obtained by fusing borax in the loop of a platinum wire until a clear vitreous mass of sodium borate is obtained. At high temperatures, this has the property of dissolving many metallic compounds, some of which produce characteristically colored borax glass beads. This borax bead is then dipped into a solution or solid to be tested, and then heated in the oxidizing flame until clear or transparent. By repeating this a number of times, the intensity of color can be increased to the desired shade, which gives the characteristic color of the metal present. In the table below is given the color of the borax bead in both the oxidizing and reducing flames.

3. Salt of Phosphorus Bead.—Salt of phosphorus beads are prepared in the same manner as borax beads; namely, by heating $\text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$ (Microcosmic Salt). The clear, colorless bead obtained is sodium metaphosphate. In the table below is given the color of the borax beads obtained in both the oxidizing and reducing flames, when the different metals are present.

4. Sodium Carbonate Bead.—The sodium carbonate bead is used only in the test for manganese compounds. The sodium carbonate is fused to a bead which is not transparent, and is then dipped into the substance to be tested for manganese and then heated in the oxidizing flame after being also dipped into KClO_3 .

The color produced, if manganese is present, is due to the formation of sodium manganate which is green when hot, and bluish green when cold.

COLOR OF FLAMES AND BEADS

ELEMENT	COLOR OF FLAME	BORAX BEAD		PHOSPHORUS SALT BEAD	
		Oxidizing Flame	Reducing Flame	Oxidizing Flame	Reducing Flame
Potassium.....	Purple-red through blue glass				
Sodium.....	Yellow				
Lithium.....	Crimson (Invisible through green glass)				
Barium.....	Yellowish-green				
Strontium.....	Scarlet (Yellow through green glass)				
Calcium.....	Orange-red (Green through green glass)				
Chromium Hot.....		Yellow	Green	Dirty-green	Dirty-green
Cold.....		Yellow-green	Green	Fine-green	Fine-green
Manganese Hot.....		Violet		Dark-violet	
Cold.....		Red-violet		Violet	
Iron Hot.....		Yellow to orange-red	Bottle-green	Brown-yellow	Red-yellow
Cold.....		Yellow	Pale green	Light yellow	
Cobalt Hot.....		Blue	Blue	Blue	Blue
Cold.....		Blue	Blue	Blue	Blue
Nickel Hot.....		Violet	Opaque-gray	Dark-red	Dirty-red
Cold.....		Red-brown	Opaque-gray	Yellow	Yellow
Copper Hot.....	Green (CuO)	Light-green	Pale-green	Green	Brown-green
Cold.....	Blue (CuCl ₂)	Blue	Opaque-red	Blue	Opaque-red
Boron Hot.....	Yellow-green (moistened with H ₂ SO ₄)				
SODIUM CARBONATE BEAD					
Manganese Hot.....		Green	Pale-green		
Cold.....		Bluish-green	Colorless		

REAGENTS

Methods of Preparation

Hydrochloric Acid 6M.—Prepared by diluting the HCl sp. gr. 1.19 in the ratio of 1:1 with water.

Hydrochloric Acid 3M.—Prepared by diluting the HCl sp. gr. 1.19 in the ratio of 1:3 with water.

Nitric Acid 6M.—Dilute HNO_3 sp. gr. 1.42 in the ratio of 1:3 with water.

Sulphuric Acid 9M.—Dilute H_2SO_4 sp. gr. in the ratio of 1:1 with water.

Acetic Acid 5M.—Dilute 285 ml of glacial acetic acid with 715 ml of water.

Ammonium Hydroxide 5M.—Dilute NH_4OH sp. gr. 0.90 in the ratio of 1:2 with water.

Ammonium Carbonate.—Dissolve 192 grams in a mixture of 80 ml of ammonium hydroxide sp. gr. 0.90 and 500 ml of water. Dilute to 1 liter. This is approximately **2M**.

The solutions of the other reagents employed are approximately 0.2N with reference to the part of the compound which is employed in the reactions for which it is used, except where otherwise designated in the directions.

1. **Alizarin Solution.**—This is a solution of alizarin containing 0.01 gram in 100 ml alcohol.

2. **Ammonium Molybdate Solution.**—Add 100 grams of MoO_3 to a mixture of 171 ml of water and 144 ml of NH_4OH sp. gr. 0.90 and stir until solution is complete. Slowly, add this solution, with constant stirring, to a solution of 489 ml HNO_3 sp. gr. 1.42 and 1148 ml of water. Allow the mixture to stand for 24 hours in a warm place, and then decant the clear solution.

3. **Calcium Sulphate.**—Saturated solution.

4. **Fehling's Solution.**—This consists of the following solutions *A* and *B* which are preserved in separate containers and mixed in equal portions just as they are to be used.

A. Dissolve 34.65 grams of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in water and dilute to 500 ml.

B. Dissolve 175 grams of Rochelle salts, $\text{KNaC}_4\text{H}_4\text{O}_6$, and 50 grams of NaOH in water and dilute to 500 ml.

5. **Dimethylglyoxime.**—Dissolve 6 grams in 500 ml of warm 95 per cent alcohol. This gives 0.1M solution.

6. **Phenolphthalein.**—A 1 per cent solution in 50 per cent ethyl alcohol.

7. **Potassium Pyroantimonate.**—Add 20 grams of the salt to 1000 ml boiling water. Boil for a minute or two, and when the salt is nearly all dissolved, cool quickly, and add about 30 ml of a 10 per cent KOH solution. Filter.

8. **Sodium Cobaltinitrite.**—Dissolve 250 g of NaNO_2 in 500 ml of water. Add 180 ml of 5N acetic acid, and 25 grams of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$. Let the mixture stand over night. Filter and dilute to one liter.

9. **Sodium Sulphide Reagent.**—Saturate 1 liter of 3M NaOH with H_2S . Add 4 grams of sulphur and 3.5 grams of NaOH.

10. **Sodium Stannite.**—This solution is prepared as needed by adding to about 2 ml SnCl_2 solution sufficient NaOH solution to just redissolve the precipitate formed. Dilute this with an equal volume of water and use as a test for bismuth.

11. **Stannous Chloride $\frac{1}{2}\text{M}$.**—Dissolve 115 grams SnCl_2 in 100 ml of 12N HCl. Dilute to 1000 ml and keep in bottles containing metallic tin.

INDEX

A

Acetate, ethyl, test for, 79
 Acetic acid, 85
 Acetylene, 77
 Acidic:
 groups in analysis, 112
 oxides, 32
 Acidimetry, 57
 Acids, 32
 Acrolein, 80
 Air, oxygen in, 25
 Alcohol:
 ethyl, 77
 methyl, 79
 Alkalimetry, 57
 Aluminum:
 detection of, 97
 test for, 99
 Aluminum sulphate, 64
 Alums, 64
 Ammonia, 52
 detection of, 101
 test for, 52, 101, 102
 Amphoterism, 69
 Analysis, 15, 23
 Analytical balance, 24
 Anode, 21
 Antimony:
 detection of, 94
 test for, 96
 Arsenic:
 detection of, 94
 test for, 96

B

Balance, 24
 Barium:
 detection of, 99
 test for, 99
 Bases, 33
 Basic:
 groups in analysis, 91
 oxides, 33
 Bath:
 air, 5

Bath (*Continued*):

 molten salt, 5
 oil, 5
 sand, 5
 steam, 5
 water, 5
 Bead tests, 120
 Bismuth:
 detection of, 94
 test for, 95
 Bleach:
 hard, 58
 soft, 59
 Boiling point, elevation of, 54
 Borates, test for, 112, 115
 Boring stoppers, 7
 Bromide, test for, 112, 114
 Bromine, 48
 Bromine compounds, 60
 Burette, 57
 Hemple, 26
 Burner, 12

C

Cadmium:
 detection of, 94
 test for, 95
 Calcium:
 detection of, 99
 test for, 100
 Caramel, 81
 Carbohydrates, test for, 80
 Carbonate, test for, 111, 114
 Cathode, 21
 Cellulose, 83
 Chemical change, 18
 Chemical equivalent, 37
 Chlorates, 59
 Chloride, test for, 43, 112, 114
 Chlorides of:
 alkali group, 43
 alkaline earth group, 44
 aluminum group, 44
 arsenic group, 45
 copper group, 46
 silver group, 47
 zinc group, 45

Chlorine, 41
 water, 58
 Chromate, 73
 test for, 74, 112
 Chromium:
 detection of, 97
 test for, 99
 Chromium trioxide, 73
 Cobalt:
 detection of, 97
 test for, 97
 Collecting gases, 8
 Colloidal systems, 53
 Conductance of solutions, 56
 Constructing apparatus, 8
 Copper:
 detection of, 94
 test for, 95

D

Decantation, 6
 Decomposition, 23
 Decrepitation, 43
 Deliquescence, 44
 Detection of:
 aluminum, 97
 ammonium, 101
 antimony, 94
 arsenic, 94
 barium, 99
 bismuth, 94
 cadmium, 94
 calcium, 99
 chromium, 97
 cobalt, 97
 copper, 94
 iron, 97
 lead, 93, 94
 magnesium, 100
 manganese, 97
 mercuric mercury, 94
 mercurous mercury, 93
 nickel, 97
 potassium, 100
 silver, 93
 sodium, 100
 strontium, 99
 tin, 94
 zinc, 97
 Determination of, chemical equivalent:
 gasometric, 38
 gravimetric, 37
 Dialysis, 53
 Diffusion, 31

Direct union, 23
 Distilling, 6

E

Electrochemical series, 22
 Electrolysis, 21
 Equations for reactions in qualitative analysis, 101
 Equivalent, chemical, 37
 Ethanol, 79
 test for, 77
 Ethylene, 76
 Eudiometer, 38
 Evaporating, 6

F

Fehling's solution, 81
 Fermentation, 81
 Ferric chloride, reduction of, 29
 Ferric iron, test for, 29
 Ferrous iron, test for, 30
 Ferrous sulphate, oxidation of, 30
 Filtration, 6
 Flame colorations, 120
 Flame test, 43
 Flash point, 78
 Freezing point, depression of, 55
 Fusion mixture, 117

G

Gasoline, 77
 Glass tubing, manipulation of, 3
 Glycerol, 80

H

Heating, 4
 Hydrochloric acid, 42
 Hydrogen, 28
 test for, 21
 Hydrogen peroxide, 36
 Hydrogen sulphide, 49, 94
 Hydrolysis, 31
 Hypochlorous acid, 58

I

Insoluble substances, 116
 Iodide, test for, 112, 114
 Iodine, 48
 Iodine compounds, 60

Iron:

- detection of, 97
- test for, 98

K

Kerosene, 77

L

Lead:

- detection of, 93, 94
- test for, 94, 95

Liquids, pouring, 3

Loew's reagent, 85

M

Magnesium:

- detection of, 100
- test for, 100

Manganates, 71

Manganese:

- detection of, 97
- test for, 72, 98

Manganese dioxide, 71

Mercuric mercury:

- detection of, 94
- test for, 96

Mercurous mercury:

- detection of, 93
- test for, 94

Metaphosphate beads, 68

Metathesis, 23

Methane, 76

Methanol, 79

Metric units, 13

Milk, sugar in, 82

Molisch reaction, 80

N

Neutralization, 34

Nickel:

- detection of, 97
- test for, 97

Nitrate, test for, 67, 112, 115

Nitric acid, 66

Nitric oxide, 66

Nitrites, 67

- test for, 111

Nitrogen dioxide, 66

Nitrous acid, 67

Nitrous oxide, 67

O

Osmosis, 55

Oxidation, 30

Oxides, 27

Oxygen:

- percentage in air, 25
- preparation, 27
- test for, 15

P

Parchment paper, 83

Perchlorates, 59

Persulphates, 65

Phosphates, 68

- test for, 115

Phosphoric acid, 68

Pipette, Hempel, 27

Plaster of Paris, 64

Potassium:

- detection of, 100
- test for, 101

Precipitate, 19

Preliminary examination of solids, 111

Prepared solution for testing for acidic radicals, 113

Properties, 14

Pyrosulphates, 65

Q

Qualitative analysis, 90

- Group classification, 91
- Group I, procedure, 93
- Group II, procedure, 94
- Group III, procedure, 97
- Group IV, procedure, 99
- Group V, procedure, 100

R

Reaction, 32

Reagent, 31

Reagents, preparation of, 122

Reduction, 28

Replacement, 23

Replacement series, 19

Rest point of balance, 25

S

Salts, 34

Schweitzer's reagent, 83

Silver:

- detection of, 93
- test for, 94

Soap, 85
 analysis of, 86
 Sodium:
 detection of, 100
 test for, 101
 Solids, transferring, 3
 Solubility, 14
 of gases, 54
 of liquids, 54
 of solids, 54
 of sulphides, 50
 Solvent, 31
 Starch, 82
 Strontium:
 detection of, 99
 test for, 100
 Substitution, 23
 Sucrose, inversion of, 81
 Sugar, in milk, 82
 Sugars, 80
 inversion of, 81
 Sulphates, 63
 test for, 62, 114
 Sulphides, test for, 111
 Sulphite, test for, 111, 114
 Sulphur dioxide, 61
 Sulphuric acid, 62
 Sulphurous acid, 62
 Synthesis, 16, 23

T

Test for:
 aluminum, 99
 ammonium, 52, 101, 112
 antimony, 96
 arsenic, 96
 barium, 99
 bismuth, 95
 borate, 112, 115
 bromide, 112, 114
 cadmium, 95
 calcium, 100
 carbohydrate, 80
 carbonate, 111, 114
 chlorate, 59
 chloride, 43, 112, 114
 chromate, 74, 112
 chromium, 99
 cobalt, 97
 copper, 95
 ethanol, 77
 ferric iron, 29
 ferrous iron, 30

Test for (*Continued*):
 hydrogen, 21
 hydrogen peroxide, 36
 hydrogen sulphide, 94
 iodide, 112, 114
 iron, 98
 lead, 94, 95
 magnesium, 100
 manganese, 72, 98
 mercury, 94, 96
 methanol, 79
 nickel, 97
 nitrate, 67, 112, 115
 nitrite, 111
 oxygen, 15
 perchlorate, 59
 phosphate, 68, 115
 potassium, 101
 silver, 94
 sodium, 101
 starch, 82
 strontium, 100
 sugars, 80
 sulphate, 62, 114
 sulphide, 111
 sulphite, 111, 114
 textile fibres, 83
 thiosulphate, 114
 tin, 97
 zinc, 98

Textile fibres, 83
 Thiosulphates, 65
 test for, 114
 Tin:
 detection of, 94
 test for, 97
 Tollen's reaction, 81

V

Vinegar, 85

W

Wash bottle, 13
 Washing precipitates, 7
 Water, 31
 Water of crystallization, 44
 Weighing, 24

Z

Zinc:
 detection of, 97
 test for, 98

RAYMOND H. FOGLER LIBRARY
DATE DUE

[illegible]



UNIV OF MAINE



3 7550 01052144 2